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# **Science & Technology**

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# Science & Technology

## USSR: Chemistry

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UDC 661.632.1:541.127:542.92(575.1)

**Dynamics of Sulfuric Acid Extraction of Kyzylkum Phosphorite Concentrate in Dihydrate Regimen**

907M0204C Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 26 Jan 89) pp 7-9

[Article by L. B. Usharova, F. M. Mirzayev, A. V. Boridko, and L. V. Dryuk; Tashkent Order of Peoples' Friendship Polytechnic Institute imeni Abu Raykhana Beruni]

[Abstract] The process of sulfuric acid extraction of a representative sample of phosphorite concentrate from the Dzheroy Mine was investigated. Its dynamics were studied as a function of temperature and  $P_2O_5$  concentration in the extraction slurry's liquid phase. It was established that at all studied temperatures and  $P_2O_5$  concentrations, the extent of  $P_2O_5$  conversion in solution (in the extraction slurry's liquid phase) increased over time, achieving a maximum at 2-6 hours depending on the  $P_2O_5$  concentration in the liquid phase and temperature. Optimum extraction conditions were determined. Figures 2; references: 2 Russian.

UDC 541.8:541.123.6:631.8

**System Diammonium Phosphate-Malonic Acid-Water**

907M0204D Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 22 May 88) pp 9-12

[Article by F. S. Askarova, A. Kh. Narkhodzhayev, and S. Tukhtayev; Institute of Chemistry, Uzbek SSR Academy of Sciences]

[Abstract] The system diammonium phosphate-malonic acid-water was studied by the direct polythermal method. On the basis of data from polytherm profiles and side-view triangular diagram polytherms, a polythermal diagram was constructed for this system's solubility in the temperature range -18.6 to 50°C. On the diagram studied, crystallization regions were determined for monoammonium phosphate, malonic acid, and ice, which covered at three triple points. Figures 2; references: 7 Russian.

UDC 543.544:54.412.2:541.49:546.9

**Concentration of Palladium, Platinum, and Rhodium by Extraction Chromatography**

907M0229A Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 4, Apr 90 (manuscript received 10 Mar 88) pp 671-677

[Article by Ye. M. Basova, V. M. Ivanov, T. A. Bolshova, and N. A. Babkova; Moscow State University imeni M. V. Lomonosov]

[Abstract] An effective method was developed for group extraction-chromatographic concentration of Pd (II), Pt (II), and Rh (III) on a column thermostatically controlled at 85° C and filled with Teflon impregnated with 1-(2-pyridylazo)-2-naphthol in isopentanol while passing aqueous acetate-sulfate solutions (pH 3.0-3.8) at a flow rate of 0.25 ml/min. Chelates were eluted from the column with 2-3 ml of a 2:1 mixture of chloroform and isopentanol. The concentrate could be analyzed spectrophotometrically or by high-performance liquid chromatography (HPLC). Figures 4; references: 10 Russian.

UDC 543.7:546.719

**Sensitivity Improvement of the Thiocyanate Method for Determining Rhenium**

907M0229B Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 4, Apr 90 (manuscript received 1 Jul 88) pp 683-686

[Article by N. M. Kuznetsova and N. G. Monakhova]

[Abstract] In this work, the effect of iron (III) and nitrate ions on rhenium thiocyanate complex formation was studied in sulfuric acid media. It was found that introducing 50-200 µg of iron into the reaction medium increased the reaction sensitivity by a factor of 1.2-1.3. Introducing 400 mg of nitrate ion into perchloric acid solution and 50-750 mg of nitrate ion into sulfuric acid solution increased the reaction sensitivity by a factor of 1.5 and reduced the lower limit for rhenium concentration determination to 0.5 µg in an aliquot portion. Figures 2; references 8: 5 Russian, 3 Western.

UDC 546.98:543.662:54.412.2

**4-Methyl-2-(2'-Oxynaphthylazo-1')thiazol and 4-Adamantyl-2-(2'-Oxynaphthylazo-1')thiazol as Reagents for Photometric Determination of Palladium**

907M0229C Moscow ZHURNAL ANALITICHESKOY KHIMII in Russian Vol 45 No 4, Apr 90 (manuscript received 6 May 89) pp 687-691

[Article by A. T. Pilipenko, Ye. A. Karetnikova, and N. A. Dyachenko; Kiev State University imeni T. G. Shevchenko]

[Abstract] Reactions of palladium (II) with 4-methyl-2-(2'-oxynaphthylazo-1')thiazol (MOT) and 4-adamantyl-2-(2'-oxynaphthylazo-1')thiazol (AOT) were studied spectrophotometrically. It was established that in aqueous organic solutions in the presence of 1 M HCl, electrically neutral complexes with the formula  $PdHRCI_2$  formed. Their chemical-analytical characteristics were determined:  $\epsilon_{738} = 1.7 \times 10^4$  ( $\epsilon$  being the molar absorption coefficient) and  $\log \beta = 19.4 \pm 0.1$  ( $\beta$  being the stability constant of the complex) (MOT);  $\epsilon_{710} = 0.7 \times$

$10^4$  and  $\log \beta = 17.2 \pm 0.1$  (AOT). A method was developed for determining the palladium concentration in industrial solutions. Figures 2; references 14: 12 Russian, 2 Western.

UDC 543.544.25

**Improvement of the Accuracy of Gas Chromatographic Analysis of Moist Gaseous Samples on Columns With Polysorb-1**

907M0229D Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45 No 4, Apr 90 (manuscript received 28 Dec 88) pp 692-695

[Article by Ye. K. Maltsev, V. Ya. Sivchenko, and N. P. Ovcharenko; Dnepropetrovsk Branch for Development of Measuring and Test Equipment, All-Union Scientific Research Institute for Mine Rescue]

[Abstract] Filling the Polysorb-1-containing portion of a column with silica gel led to chromatographic band smearing due to water and resulted in a reduced distorting effect of water signals on the results of gas chromatographic analysis of moist gaseous samples. The accuracy of results from determining components without filter-drying the sample increased more than fourfold in this case. Figures 1; references: 2 Russian.

UDC 543.544:547.922

**Gas Chromatographic Determination of Cholesterol in Blood Plasma**

907M0229G Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45 No 4, Apr 90 (manuscript received 18 Oct 88) pp 802-808

[Article by G. M. Kheyfets; Scientific Research Institute of Experimental Medicine, USSR Academy of Medical Sciences, Leningrad]

[Abstract] Total cholesterol determination in blood plasma by gas-liquid chromatography could be conducted in one step by combining cholesterol ester methanolysis and cholesterol extraction. While determining free cholesterol in blood plasma, it was possible to combine cholesterol extraction and selective solvolysis of nonvolatile phospholipids and slightly volatile triglycerides to rid the sample of their excess amounts. References 18: 4 Russian, 14 Western.

UDC 543.422.546.87.42.41.56

**Atomic Absorption Analysis of the High-Temperature Superconducting System Bi-Sr-Ca-Cu-O**

907M0229H Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45 No 4, Apr 90 (manuscript received 1 Jun 89) pp 809-811

[Article by N. K. Belskiy, L. I. Ochertyanova, and Yu. I. Krasilov; Institute of General and Inorganic Chemistry imeni N. S. Kurnakov, USSR Academy of Sciences, Moscow]

[Abstract] For the high-temperature superconducting system Bi-Sr-Ca-Cu-O, the mutual effects of elements were studied during their atomic absorption determination in an air-acetylene flame and in an electrothermal atomizer. Mutual effects were eliminated during flame analysis by adding NaCl. The relative standard deviation for flame determination of elements was 0.007 for Bi, 0.004 for Ca, and 0.002 for Sr and Cu. In electrothermal atomic absorption, the corresponding values were 0.02, 0.03, and 0.015. References 4: 2 Russian, 2 Western.

UDC 543.544.45.541.6.547.26

**Model of Aliphatic Alcohol Molecular Connectivity and Chromatographic Retention**

907M0229I Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45 No 4, Apr 90 (manuscript received 15 Jun 89) pp 812-814

[Article by A. V. Volkov and V. M. Nabivach; Dnepropetrovsk Chemical Engineering Institute]

[Abstract] It was shown that models of the molecular connectivity of aliphatic alcohols, formed on the basis of chain indexes and branching of the first four orders, accurately described their gas chromatographic retention characteristics. The two- and four-factor equations derived could be used to predict retention indexes of aliphatic alcohols and to identify them in complex mixtures without a standard. References 13: 2 Russian, 11 Western.

UDC 543.4:543.7

**Photometric Determination of 3,5-Dinitroaniline**

907M0229J Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45 No 4, Apr 90 (manuscript received 17 Apr 89) pp 815-817

[Article by A. A. Mokhov (deceased), L. B. Leontyeva, and N. P. Mukhovikova; Leningrad Technological Institute]

[Abstract] In this work, a method for photometric determination of 3,5-dinitroaniline (DNA) in the presence of 3,5-dinitrobenzoic acid and 3,5-dinitrobenzazide impurities was developed. The method was used to analyze industrial samples with a DNA content of 90-95%. The relative standard deviation did not exceed 0.03. Figures 1; references: 4 Russian.



UDC 547.826+66.092

**Effect of Passivation on Catalytic Properties of Alloyed Al-Ni-Ti Catalyst**

907M0204K Tashkent UZBEKSKIY KHIMICHESKIY  
ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript  
received 16 Dec 88) pp 52-54

[Article by T. A. Kirgizbayev, V. Kh. Kaysas, M. Kh. Yakubova, A. Yu. Korontsevich, K. K. Karatayev, and M. F. Abidova; All-Union Scientific Research Chemical Engineering Institute for the Medical and Microbiological Industry]

[Abstract] The interconnection of the passivation method, catalytic properties, and structure of an aluminum-nickel-titanium catalyst was determined. An industrial pyrophoric catalyst was passivated by various methods with the aim of improving the process's technological effectiveness. With the use of physical chemical methods, it was established that passivated samples had identical phase compositions but differing morphological patterns. The presence of NiO stabilized metallic nickel centers (on which the catalytic process occurred) in the active state. References 6: 5 Russian, 1 Western.

UDC 66.061.352

**Phosphoric Acid Extraction by Tributyl Phosphate From Karatau Phosphorite Extraction Phosphoric Acid**

907M0204L Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 25 Nov 88) pp 54-57

[Article by T. V. Tokmakova, R. Ya. Yakubov, B. T. Kunin, V. G. Moshkova, and L. V. Konyakhina; Voskresenskoye Branch, Scientific Research Institute for Fertilizers and Insectofungicides imeni Professor Ya. V.

Samoylov, Minudobreniya (Mineral Fertilizers) Scientific Production Association]

[Abstract] The distribution of phosphoric acid between the aqueous and organic phases in the system EPA (extraction phosphoric acid from Karatau phosphorites)-TBP (tributyl phosphate)-kerosene at 293-343 K was studied. An empirical dependence of the equilibrium concentration of phosphoric acid in the organic phase on its equilibrium concentration in the aqueous phase, on the TBP concentration in the extractant, and on temperature was established. The feasibility of using the results presented for determining technological parameters of extracting phosphoric acid from EPA with tributyl phosphate was shown. References 5: 4 Russian, 1 Western.

UDC 541.135

### Hydrogen Overvoltage on Gold Electrode in Phosphate Solutions

907M0100A Kiev UKRAINSKIY KHIMICHESKIY  
ZHURNAL in Russian Vol 55 No 11, Nov 89  
(manuscript received 19 Jul 88) pp 1165-1167

[Article by V. S. Kublanovskiy, K. I. Litovchenko, V. I. Miroshnichenko, and V. N. Nikitenko, General and Inorganic Chemistry Institute, Kiev]

[Abstract] Acidic and moderately acidic cyanoaurate electrolytes are useful for gold plating electronic components. Being less toxic than cyanides, they may be used at higher current densities at room temperature and they do not contain substances harmful to the properties of photoresistors and the plastic bases of printed circuits. Reduction of gold from dilute weakly acidic electrolytes is accompanied by hydrogen evolution, and it is of both practical and theoretical interest to determine the effects of each component of the gold plating electrolyte on the hydrogen overvoltage on a gold electrode. In the present work polarization curves were determined for a 2 cm<sup>2</sup> gold electrode. The working solutions contained 20 grams per liter NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, 40 grams per liter (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>, and either 5 mg per liter lead carbonate and/or 1 gram per liter hydrazine sulfate at pH 5.6 adjusted with phosphoric acid. Addition of the lead carbonate alone had no noticeable effect on hydrogen reduction, while a joint presence of both lead carbonate and hydrazine sulfate shifted the electrode potential to the positive side, lowered the hydrogen overvoltage, and lowered the energy barrier for the electrochemical reduction of hydrogen. Figures 3; references 4 (Russian).

UDC 541.135.6:621.357.1

### Polarization Nature of Pseudoliquified Activated Charcoal-Based Electrodes

907M0149C Kiev UKRAINSKIY KHIMICHESKIY  
ZHURNAL in Russian Vol 56 No 1, Jan 90 pp 33-36

[Abstract of article by K. A. Kazdobin, L. A. Klimenko, and N. A. Shvab]

[Abstract] The polarization of activated charcoal-based pseudoliquified electrodes was studied. Granules of charcoal 0.8-1.0 mm in size obtained from synthetic raw material were used as the electrode material, which either contained nitrogen or was promoted with palladium and tungsten carbide. Potential characteristics were measured as a function of the polarization potential of a platinum current collector, the electrolytic flow rate, and polarization time. The height of the quiescent layer was 10 mm. In order to obtain reproducible values for the stationary electrode potentials, the electrode materials were kept in contact with a working solution of 0.05 M NaCl, a phosphate buffer with 7.4 pH, and a 1:1 hydrolysis solution in a phosphate buffer. The electrode potentials were measured by potentiostatic probing on a

P-5827M potentiometer. The polarization potential interval was +0.9 to -0.9 V. It was shown that the value of the electrode potential of granulated electrode materials can be kept between 0.8 and -0.8 V, depending on the processes occurring inside each granule, primarily the electroreduction of electrochemically active gases. Electroreduction of oxygen in a diffusion regimen was used to show that the effective working surface of these types of electrodes is practically the same as its visible surface, i.e., little current is expended to polarize the surface of the granule pores. Figures 3, table 1; references 6: 5 Russian, 1 Western.

UDC 532.77

### Relationship Between the Viscosity and Electrical Conductivity of Aqueous Solutions of Ammonium Thiocyanate

907M0204E Tashkent UZBEKSKIY KHIMICHESKIY  
ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript  
received 16 Dec 88) pp 19-20

[Article by M. M. Alimova, F. M. Mirdzhalalov, and S. M. Mezheritskiy; Tashkent Order of Peoples' Friendship Polytechnic Institute imeni Abu Raykhana Beruni]

[Abstract] The relationship between  $\nu$  (viscosity) and  $\chi$  (specific electrical conductivity) was determined by measuring the kinematic viscosity and specific electrical conductivity of aqueous solutions of ammonium thiocyanate in the temperature range 20-90°C and at concentrations of 10-30%. An empirical formula expressing this relationship was obtained:  $\nu_s/\nu_w = 0.747 + 1.262\chi - 0.829\chi^2$ , where  $\nu_s$  is the viscosity of the solution and  $\nu_w$  is the viscosity of water. The relationship was found to be independent of temperature and concentration in the ranges studied. Figures 2; references: 5 Russian.

UDC 541.18:651.185.1

### Flocculating Activity of Ionene Polymers Based on Products of Reaction of Epichlorohydrin With Polyamines

907M0204F Tashkent UZBEKSKIY KHIMICHESKIY  
ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript  
received 23 Dec 88) pp 23-26

[Article by N. Ye. Yedgorov and A. T. Dzhalilov; Tashkent Order of Peoples' Friendship Polytechnic Institute imeni Abu Raykhana Beruni]

[Abstract] New water-soluble cationic polyelectrolytes, obtained by polymerizing epichlorohydrin with polyamines, were synthesized and studied. Their physicochemical properties and the behavior of their aqueous solutions were studied. The polymers obtained were studied as flocculants in the process of precipitating solid phases from suspensions and as flotation activators in processes of recovering tungsten from industrial slurries and solutions. It was shown that the cationic polyelectrolytes synthesized were highly effective in the role of flocculants. Figures 2; references: 3 Russian.

UDC628.312:547.1(62-713)

**Stabilizing Effects of Organic Substances in Municipal Wastewaters on Crystallization of Calcium Carbonate in Recirculating Water of Cooling Systems**

907M0224A Kiev *KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12 No 4, Apr 90 (manuscript received 11 Aug 89) pp 312-315*

[Article by A. K. Khachaturov, I. N. Rozhdov, and I. A. Malakhov, Azerbaijan Institute of Petroleum and Chemistry imeni M. Azizbekov, Baku; Polytechnical Institute, Novocherkassk]

[Abstract] An analysis was conducted to determine the effect of organic substances on the degree of supersaturation of  $\text{CaCO}_3$  in municipal wastewaters in order to assess the degree of evaporation that can be tolerated in cooling systems before deposits become a problem. The study involved analysis of the relationship between the stabilizing effects of organic substances and chemical oxygen demand and the relationship between temperature and ionization constants. Incorporation of the data into the Davis equation for coefficients of activity led to an equation useful for determination of evaporation coefficients under the applicable conditions. Figures 2; tables 1; references 12: 10 Russian.

UDC541.123.7:547.392.4:622.765/793

**Calcium and Iron Ions and Surfactants in Recirculating Waters in Apatite Flotation**

907M0224B Kiev *KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12 No 4, Apr 90 (manuscript received 7 Jul 89) pp 315-322*

[Article by V. K. Karzhavin, Kola Scientific Center, USSR Academy of Sciences; Geological Institute, Apatity]

[Abstract] An analysis was conducted of the calcium and iron ions and surfactants used in apatite flotation in order to assess precipitation kinetics and maintain the selectivity of the flotation process. Calculations and experimental data demonstrated that treatment of the recirculating water involves marked changes in the acid-base and redox characteristics of the water predicated on the interaction of the surfactants with the ions. Accordingly, it was shown that pH and Eh may be used in monitoring water composition in apatite flotation for selection of optimum water-treatment processes. Figures 3; references 18: 8 Russian, 10 Western.

UDC622.793.5

**Precipitation of Sulfates From Waste Waters by Calcium Aluminates**

907M0224C Kiev *KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12 No 4, Apr 90 (manuscript received 12 Sep 89) pp 319-322*

[Article by Ye. O. Salnikova, I. V. Gofenberg, Ye. N. Turanina, and L. Ye. Sitchikhina, Ural Scientific Research and Engineering Institute of the Copper Industry, Sverdlovsk]

[Abstract] Detailed studies were conducted on calcium aluminates for removal of sulfates from wastewaters. It was demonstrated that precipitation of virtually insoluble calcium hydrosulfoaluminates was best accomplished at a pH level between 11.8 and 12.1. In addition,  $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 19\text{H}_2\text{O}$  (equilibrium pH, 11.9) was shown to be the most efficient precipitating agent. Figures 2; tables 1; references 5 (Russian).

UDC543.314/.315:628.16.067

**Comparative Evaluation of Turbidity and Colloid Index as Control Parameters of Water Clarification Before Reverse-Osmosis Desalinization**

907M0224D Kiev *KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12 No 4, Apr 90 (manuscript received 17 May 89; in final form 4 Nov 89) pp 322-325*

[Article by Yu. G. Frolov and I. M. Mikerova, Moscow Institute of Chemical Technology imeni D. I. Mendeleev]

[Abstract] The colloid index and turbidity were analyzed for their utility in monitoring water clarification prior to desalinization by reverse osmosis. The calculations and experimental data demonstrated that the colloid index provided a reliable indication of the fouling characteristics of water when the turbidity was  $<0.3 \text{ TE/F}$ . In situations where turbidity is  $>0.3 \text{ TE/F}$  the colloid index approaches its upper limit and loses its relationship to the degree of water pollution and, hence, its usefulness. Changes in the colloid index should be monitored daily since turbidity is an unstable characteristic of drinking water. Finally, efficiency of the filtration system is best monitored on the basis of turbidity. Figure 1; table 1; references 8: 5 Russian, 3 Western.

UDC628.543

**Treatment of Waste Waters Loaded With Aluminosilicates**

907M0224E Kiev *KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12 No 4, Apr 90 (manuscript received 12 Sep 89) pp 365-367*

[Article by N. N. Krupina, Biotekhnika All-Union Scientific Research Institute, Grodno Branch]

[Abstract] The process of producing synthetic zeolites is accompanied by the production of large quantities of alkaline waste waters loaded with aluminosilicates, which present unique water treatment problems. Experimental studies demonstrated that treatment of such wastewaters with fresh sodium silicate and calcium hydroxide under proper conditions can lead to elimination of 99.8% of the aluminum oxide and 94% of the silicon oxide. Treatment was equally efficient whether carried out at room temperature or at 40-60°C. Figure 1; table 2; reference 1 (Russian).

UDC628.35

#### Efficient Elimination of Nitrogen Compounds in Multicompartment Aeration Tanks

907M0224F Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 12 No 4, Apr 90 (manuscript received 5 Jun 89) pp 370-373

[Article by V. B. Vasilyev and V. A. Vavilin, Institute of Water Problems, USSR Academy of Sciences, Moscow]

[Abstract] Several multicompartment aeration tank modalities were tested for their efficiency in removing nitrogen compounds in wastewaters by activated sludge. The results demonstrated that the best results were obtained with the Bardenpho process. Optimization of the parameters demonstrated that with an increase in organic load in the water the aerobic zone of the aeration tank increases and the anaerobic zone diminishes. In addition, an increase in the recirculation coefficient of active sludge enhances elimination of the nitrogen compounds in direct relationship to the organic load. Figure 1; tables 2; reference 1 (Russian).

UDC541.135:628.337:628.347

#### Operation of Electrolyzers in Electrochemical Regeneration of Aluminum-Based Coagulants

907M0224G Kiev *KHIMIYA I TEKHOLOGIYA VODY* in Russian Vol 12 No 4, Apr 90 (manuscript received 8 Aug 89) pp 373-377

[Article by Yu. V. Yepifanov and Ye. S. Matskevich, Institute of Colloid Chemistry and Water Chemistry imeni A. V. Dumanskiy, Ukrainian SSR Academy of Sciences, Kiev]

[Abstract] An electrochemical method has been developed for the treatment of natural and wastewaters involving regeneration of aluminum-based coagulants. Electrochemical regeneration of the water-soluble aluminum compounds involves relatively simple and inexpensive apparatus and equipment. In view of the repeated use of the apparatus and reusable coagulants, the system as described is suitable for treatment of 568 m<sup>3</sup> per day. Implementation of such technology should

reduce the cost of treating 1 m<sup>3</sup> of water by 5 to 10 kopecks, which is equal to a saving of 15 to 20 thousand rubles annually. Tables 3; references 16: 15 Russian, 1 Western.

UDC 543.3:546.56.546.74.546.47.546.76

#### Sorption X-Ray Fluorescent Determination of Copper, Nickel, Zinc, and Chromium in Waste Water

907M0229E Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45, No 4, Apr 90 (manuscript received 19 Jan 89) pp 766-771

[Article by N. I. Shcherbinina, G. R. Ishmiyarova, I. Ye. Nikitina, G. V. Myasoyedova, L. A. Polosukhina, I. I. Uralov, A. D. Simonova, and Ye. Ya. Danilova; Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy, USSR Academy of Sciences, Moscow; All-Union Scientific Research Institute for Water Conservation, Kharkov]

[Abstract] For x-ray fluorescent determination of copper, nickel, zinc, and chromium (III, VI) in waste water, it was proposed to use POLIORGS VII M fibrous complex-forming sorbent with amidoxime groups to preconcentrate the metals. The metals were sorption concentrated on a filter made of nonwoven material while repeatedly pumping the analyzed solution through the filter-sorbent with a peristaltic pump. An optimum sorption concentration regime was selected. The mutual effect of the metals was studied during sorption and determination. A method was developed for sorption x-ray fluorescent determination of Cu, Ni, Zn, and Cr (III, VI) in waste water. The relative standard deviation for analyzing water with a metal concentration of 0.05-1 mg/l was 0.03-0.15. Figures 4; references 15: 10 Russian, 5 Western.

UDC 546.38:546.175:534.257

#### Potentiometric Determination of Nitrate Ion in Chemical Plant Waste Water

907M0229F Moscow *ZHURNAL ANALITICHESKOY KHIMII* in Russian Vol 45 No 4, Apr 90 (manuscript received 7 Apr 89) pp 773-776

[Article by V. G. Derkasova, L. A. Moskalenko, and G. I. Kvadyayeva; Tomsk Polytechnic Institute]

[Abstract] A direct potentiometric method was developed for determining nitrate concentrations in chloride-sulfate-carbonate waste water from chemical plants by using the EM-NO<sub>3</sub>-01 domestically produced nitrate-selective electrode. NO<sub>3</sub><sup>-</sup> could be determined at concentrations ranging from 0.00062 to 10.0 g/l with a relative standard deviation not exceeding 0.1. The systematic error was negligible. Figures 1; references 16: 8 Russian, 8 Western.

UDC 54-31.537.72

**Synthesis and Physical Chemical Study of Compounds  $\text{BaCoO}_{3-x}$  and  $\text{SrCoO}_{3-x}$** 

907M0128A Moscow ZHURNAL

NEORGANICHESKOY KHIMII in Russian

Vol 35 No 1, Jan 90 (manuscript received 12 May 89)

[Article by O. V. Godzguteva, N. V. Porotnikov, G. Ye. Nikiforova, and E. A. Tishchenko, Fine Chemical Technology Institute imeni M. V. Lomonosov, Moscow]

[Abstract] A systematic study was made of reactions in the subsolidus oxide region of the title compounds. The oxides have a rhombic perovskite structure. Stoichiometry with respect to oxygen was determined, and the relationship of specific bulk electric conductivity to temperature and oxygen coefficient of the rhombic phases was studied. Figures 3; references 7: 2 Russian, 5 Western.

UDC 541.123+546.562+537.312.62

**Interfacial Correlations in Partial Cuts of System  $\text{Y}_2\text{O}_3\text{-BaO-CuO}(\text{Cu}_2\text{O})$** 

907M0128C Moscow ZHURNAL

NEORGANICHESKOY KHIMII in Russian

Vol 35 No 1, Jan 90 (manuscript received 24 Feb 89) pp 223-227

[Article by G. A. Mikirticheva, V. I. Shitova, O. G. Chigareva, S. K. Kuchayeva, L. Yu. Grabovenko, and R. G. Grebenshchikov, Silicate Chemistry Institute imeni I. V. Grebenshchikov, Leningrad]

[Abstract] Physical chemical analysis of the title system is encumbered with many experimental and methodological difficulties owing to the valence change in copper with heating, vigorous interaction of the barium compounds with the crucible material, the presence of unstable nonequilibrium phases, as well as incongruent melting. In the present work hardening-annealing, differential thermal analysis and x-ray and crystal optical methods were used to construct polythermal cuts of two sections of the title system at 850-1,450°C. Equilibrium phase relationships in the cuts are complex, passing from eutectic to peritectic. Nonequilibrium interfacial relations were detected in the system  $\text{BaCuO}_2\text{-Y}_2\text{O}_3$  in the low-temperature region as well as above the solidus line, apparently due to the chemical composition of the liquid phase. Figures 3; references 5: 2 Russian, 3 Western.

UDC 541.123.31

**Use of Formates in Synthesis of High-Temperature Superconducting Materials**

907M0128D Moscow ZHURNAL

NEORGANICHESKOY KHIMII in Russian

Vol 35 No 1, Jan 90 (manuscript received 25 Apr 89) pp 237-241

[Article by S. M. Portnova, Yu. I. Krasilov, N. T. Kuznetsov, and I. V. Balakayeva, General and Inorganic Chemistry Institute imeni N. S. Kurnakov, Moscow]

[Abstract] Discovery of high-temperature superconducting phases of the type  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  ushered in the task of developing materials having stable properties. Synthesis methods must be developed that can provide high chemical uniformity and dispersion of materials while maintaining strict phase stoichiometry. Changing over from solid phase technology to aqueous solutions modifies these difficulties to some extent; however, phase diagram research is still required. In the present work aqueous systems containing yttrium, barium, and copper formates were studied. Solubility and solid-phase composition data at 25 degC were determined and the formation of a new congruently soluble compound confirmed. A method for synthesizing ultradispersed phases that is useful as a basis for preparing high-temperature superconducting materials is presented. Figures 2; references 10: 6 Russian, 4 Western.

UDC 546.881'5.431

**Phase Composition of System  $\text{BaO-V}_2\text{O}_5$** 

907M0128E Moscow ZHURNAL

NEORGANICHESKOY KHIMII in Russian

Vol 35 No 1, Jan 90 (manuscript received 5 Apr 89) pp 253-255

[Article by B. G. Golovkin and L. V. Kristallov, Ural Department, Chemistry Institute, Sverdlovsk]

[Abstract] The phase composition of the title system and the properties of barium vanadate have been reported previously. In the present work  $\text{Ba}_3\text{V}_4\text{O}_{13}$  and a new vanadate,  $\text{Ba}_4\text{V}_2\text{O}_9$ , were synthesized from BaO and  $\text{Ba}_3\text{V}_2\text{O}_8$  at 700°C and with 80 hours of calcining time. The new vanadate crystallizes in tetragonal syngony and reacts with air carbon dioxide at 800-1,000°C to form a carbonate and an orthovanadate. IR-spectra and x-ray data are presented. Figure 1; references 11: 7 Russian, 4 Western.

UDC 541.183.12

**Amphoteric Properties of Titanium and Zirconium Phosphate-Based Ionites With Low Phosphorus Content**

907M0149A Kiev UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 56 No 1, Jan 90 pp 7-10

[Abstract of article by A. I. Bortun, G. A. Malinovskiy, S. A. Khaynakov, and V. N. Belyakov; Institute of General and Inorganic Chemistry, Ukrainian Academy of Sciences, Kiev]

[Abstract] The authors studied the anion-exchange properties of inorganic cationites based on titanium and zirconium phosphates with low phosphorus content. The influence of the chemical composition and aging of the ionite, the nature of the anion, and the acidity of the medium on the sorption efficiency of the anions were of particular interest. A gel method was used to synthesize

the ionites. Variation of the  $P:Me^{IV}$  molar ratio in the sorbent matrix was controlled by regulating the flow of the starting reagents into the mixer. The hydrogel granules were rinsed in demineralized water to achieve a pH of 3.0-3.5 and air-dried, and specimens with different residual moisture contents were selected at specific intervals. Anion-exchange properties were evaluated from  $PO_4^{3-}$ ,  $SO_4^{2-}$ , and  $Cr_2O_7^{2-}$  ion sorption from 0.05 N solutions of the appropriate acids and their nitrate salts. The phosphate and bichromate ion content in the solutions was determined spectrophotometrically, and the sulfate ion content was determined nephelometrically. The results showed that low-phosphorus titanium and zirconium phosphates have pronounced amphoteric properties due to the presence of functional phosphate and hydroxyl groups in their structure. The anion-exchange capacity is 2-4 mgEq/g, and the cation-exchange capacity is 3-5 mgEq/g. The "phosphorus-deficient" cationites manifested anion-exchange properties; their sorption activity was determined by chemical content. As solution acidity decreased, the anion-exchange capacity of all the cationites studied diminished to 30-40% of its original value at pH 7. Figures 4; references 13: 10 Russian, 3 Western.

UDC 541.183

#### Acid-Base Properties Photochemically Active Titanium Oxide Surfaces

907M0149B Kiev UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 56 No 1, Jan 90 pp 10-14

[Abstract of article by N. D. Konovalova, V. I. Stepanenko, A. V. Fesenko, and V. M. Ogenko]

[Abstract] Programmed thermodesorption was used to study the acid-base properties of the surface of  $TiO_2$ ,  $TiO_2$  (10%)/ $SiO_2$ ,  $TiO_2$  (1%)/ $SiO_2$ , Ag (1%)/ $TiO_2$ , Pd (1%)/ $TiO_2$ , and  $SiO_2$ . Titanium dioxides were obtained through oxidation pyrolysis of titanium chloride or a titanium chloride-silicon chloride mixture. The titanium dioxide was applied to the surface of pyrogenic silicon from a 1% solution of tetrabutoxytitanium in butanol-1 with subsequent steam hydrolysis. Silver and palladium were applied to the surface of the titanium dioxide from a 1% solution of aqueous solutions of nitrate and chloride, with subsequent irradiation by a mercury vapor lamp. The specific specimen surface was determined from argon adsorption. The specimens were heated through at 823 K and cooled in a helium stream to the 308 K adsorption temperature prior to each adsorption-desorption cycle. The photochemical activity of the oxides was determined by irradiating them with UV light at 353 K during the oxidation reactions of the hydrocarbons with atmospheric oxygen. The acid-base properties were compared with these data. Gas chromatography was used to study thermodesorption and adsorption.

Ammonia, carbon dioxide, and methane molecules were used as test molecules. The quantity of desorbing gases was recorded by a katharometer. It was shown that the quantitative surface acid characteristics were much higher for the homogeneous  $TiO_2/SiO_2$  mixture and for the  $TiO_2$  with the Ag and Pd applied to the surface. The oxides' photochemical activity sequence correlates with the quantity of carbon dioxide chemisorbed on a unit of oxide surface. Figure 1, table 1; references 9 Russian.

UDC 541.127:541.183.5

#### Kinetics of Metal Ion Sorption From Sea Water on Clinoptilolite Sorbents

907M0158A Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKAYA in Russian No 2, Feb 90 (manuscript received 7 Dec 88) pp 263-267

[Abstract of article by R. Kh. Khamizov, T. Yu. Butenko, M. L. Veber, and Ye. V. Zaytseva; Institute of Geochemistry and Analytical Chemistry, imeni V. I. Vernadskiy, USSR Academy of Sciences, Moscow, and Institute of Chemistry, Far Eastern Center, USSR Academy of Sciences, Vladivostok]

[Text] A comparative study was done on the kinetics of metal ion sorption from sea water on two types of sorbents with the same granulation: a natural Tedzami clinoptilolite and a synthetic bidispersed sorbent. The natural "T" specimens had a radial grain size of 0.1-0.25 mm for  $T_1$  and 0.5-1 mm for  $T_2$ . The synthetic 51% powdered "TG" clinoptilolite specimens were prepared in an ice bath by adding pulverized clinoptilolite to an aqueous solution of 50% acrylamide and 5%  $N,N'$ -methylenebisacrylamide in a quantity equal to the solution mass and by continuously agitating the suspension as the polymerization initiators were added. The polymer was cut up into granules, and fractions with the desired particle size were winnowed out. The sorbents were placed in 100-g quantities into graduates ( $S=130\text{ cm}^2$ ,  $l=30\text{ cm}$ ). A fluidized bed of sea water was passed through at  $>1,000\text{ sp vol/h}$ . The specimens were then removed, quickly rinsed, and regenerated with a 0.5-M solution of  $NH_4NO_3$  at  $1\text{ sp vol/h}$  in the same size graduates. The zeolites were converted into original ionic form with a 2-M solution of  $NH_4NO_3$ . Control for complete conversion and regeneration was determined from the absence of  $K^+$  ions in the filtrate. Atomic absorption was used to measure metal ions in the regenerated solutions. X-ray spectral analysis was used to analyze the specimens before, during, and after regeneration. Sorption speed is basically limited by diffusion in the zeolite microgranules, which are typically 15-20  $\mu\text{m}$  in diameter, i.e., practically commensurate with the 15-20  $\mu\text{m}$  single crystals of the clinoptilolite specimen used. Figures 4; references 9: 8 Russian, 1 Western.

UDC 541.128.34:541.49:546.97:541.64:542.941.7:547.546

**Structure and Catalytic Activity of Carrier-Affixed Metal Complexes. Communication 3. Rhodium Complexes Affixed to Modified Polymers and Their Catalytic Properties in Reduction Reactions of Nitrobenzene With Chemically Bonded Hydrogen**

907M0158B Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKAYA in Russian No 2, Feb 90 (manuscript received 11 Jul 88) pp 268-273

[Abstract of article by V. F. Dovganyuk, V. Z. Sharf, V. K. Belyayeva, I. N. Marov, Zh. L. Dykh, L. I. Lafer, and V. I. Yakerson; Institute of Organic Chemistry imeni N. D. Zelinskiy, USSR Academy of Sciences, Moscow, and Institute of Geochemistry and Analytical Chemistry imeni V. I. Vernadskiy, USSR Academy of Sciences, Moscow]

[Text] Heterogenized rhodium complex-based catalysts were synthesized. The polymer carriers were made by aminating chloromethylated styrene copolymer with divinylbenzene, 3(5)-methylpyrazene, or imidazene or by copolymerizing N-vinylbenzylimidazene with divinylbenzene. To synthesize the catalysts, 2 g of carrier was placed in a Wolff bottle, to which was added 40 ml of a 0.01 M solution of  $\text{RhCl}_3$  in MeOH. The reaction proceeded in boiling solvent in an argon atmosphere for 1-3 hours with constant agitation. The rhodium content was photometrically measured from the attenuation of the concentration of complex in the solution. "Homogeneous" analogues were synthesized by the interaction of  $\text{RhCl}_3$  and the appropriate heterocyclic amine in an MeOH solution. The Rh:amine molar ratio was 1:10. The complex formed during the reaction was precipitated, filtered, rinsed with alcohol and ether, and air-dried. Measured quantities of the catalyst and  $\text{NaBH}_4$  were placed in a test tube. Propanol-2 was added, heated to boiling (82°), and held for 15 minutes. After the system was cooled, the solvent was removed, and the catalyst was dried (30-40°). All the steps were performed in argon. The system was evacuated, the specimen transferred to the quartz ampule soldered to the test tube, and the ampule vacuum-sealed. The carrier and complex specimens were made by pressing them with KBr. The solid-substance specimens were suspended in petroleum jelly and placed between CsI plates. A Radiopan SE/X2544 spectrometer was used to take EPR spectra of the original (in air) and heterogenized (in vacuum) Rh complexes. IR spectra in the 3600-700  $\text{cm}^{-1}$  region were taken with UR-20 and Specord M-80 spectrometers. Between 700 and 200  $\text{cm}^{-1}$ , a Perkin-Elmer was used. A Specord M-40 was used to record UF spectra in the visible region by using the diffuse reflection method. The complex is formed by coordination of the Rh with "pyridene"-type nitrogen atoms. Partial reduction of Rh(III) occurs during complex formation and is significantly conditioned by interaction with the heterocyclic amine molecule. The catalyst specimens are reactive in the reduction reaction only after treatment with  $\text{NaBH}_4$

in the propanol-2 solution. Without the catalyst,  $\text{NaBH}_4$  in the propanol-2 solution does not reduce nitrobenzene. Figures 2, tables 1; references 19: 8 Russian, 11 Western.

UDC 541.128.34:541.27:549.67

**Catalytic Activity and Acidic Properties of Al-Pentasil and Isomorphically Substituted (Fe,B) Silicates**

907M0158C Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKAYA in Russian No 2, Feb 90 (manuscript received 2 Nov 88) pp 273-277

[Abstract of article by T. V. Vasina, T. R. Bruyeva, Ye. G. Khelkovskaya-Sergeyeva, G. I. Kapystin, B. K. Hefedov, A. L. Klyachko, and O. V. Bragin; Institute of Organic Chemistry imeni N. D. Zelinskiy]

[Text] A study was done to determine the link between the acidic characteristics and catalytic properties of TsVM ( $\text{SiO}_2/\text{Al}_2\text{O}_3(\text{M})=35$ ), pentasil-280 ( $\text{M}=280$ ), and silicalite Al-pentasil and Fe- ( $\text{M}=234$ ) and B-silicates ( $\text{M}=179$ ), all with pentasil structures, in aliphatic hydrocarbon (normal hexane, octane, and decane of 98% or greater purity) conversion reactions. Only  $\text{Na}_2\text{O}<0.1\%$  H-forms were studied. The catalytic tests were done at 350 and 450° in a flow reactor on specimens preactivated in flowing air for 5 hours at 550°. The volume of the catalytic charge was 2.5  $\text{cm}^3$ . The reagent volume velocity was 0.9  $\text{h}^{-1}$ . The experiment lasted 3 hours, during which the original reactivity of the catalysts (which were regenerated for 2 hours between tests) remained practically unchanged. To study zeolite acidity, the heat of  $\text{NH}_3$  adsorption at 30° was measured with a Kalve-type microcalorimeter connected to a vacuum apparatus. Catalytic activity was found to be directly dependent on acidity. The silicates are less acidic and much less active in cracking and aromatization of the normal alkanes than are the Al-pentasil. Pentasil with a lower concentration of acidic centers had a lower degree of alkane conversion, and the aromatization ability of the zeolites dropped sharply. Although they have different acidity characteristics, the Fe- and B-silicates have about the same cracking and aromatization activity. On the Fe-silicate,  $\text{NH}_3$  is adsorbed at higher heats both on the acid centers active in these reactions and on the inactive  $\text{Fe}_2\text{O}_3$  phase. Figures 2, tables 1; references 17: 11 Russian, 6 Western.

UDC 541.124:541.128:542.92:547.592.1-39

**Acid-Catalyzed Disassociation of Hydroperoxides in Presence of Ketones**

907M0158D Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKAYA in Russian No 2, Feb 90 (manuscript received 21 Oct 89) pp 278-285

[Abstract of article by L. V. Petrov, T. I. Drozdova, L. Ya. Lyuta, and V. M. Solyanikov; Department of the



Institute of Physical Chemistry imeni N. N. Semenov,  
USSR Academy of Sciences, Chernogolovka]

[Text] A study was done to gain a clearer understanding of how disassociation products influence the disassociation reaction of hydroperoxides. Cyclohexylhydroperoxide (HPCH) was synthesized by oxidizing vacuum-distilled solutions of cyclohexane containing 75 and 90% HPCH, with cyclohexanol practically the only impure component. The gross hydroperoxide disassociation speed was determined iodometrically. The disassociation products (cyclohexanol and cyclohexanone) were analyzed chromatographically at 323 K after first breaking down the residual HPCH in the samples by adding a quantity of triphenylphosphine two times greater than the quantity of hydroperoxide. The quantity of cyclohexanol formed during the course of the kinetic experiment was determined by combining the chromatographic and iodometric data. It was found that the ketones and aldehydes accelerate the disassociation of hydroperoxides in a solution of acetonitrile in the presence of strong acids. This effect is associated with the formation of polyperketals (polyperacetals) subject to rapid acid-catalytic disassociation. For the first time, the acid-catalyzed disassociation reaction of cyclohexylhydroperoxide was shown to be macrophasal. The initially slow disassociation proceeds homolytically and yields cyclohexanone as a product, which speeds up the disassociation. Figures 4, tables 4; references 7: 6 Russian, 1 Western.

UDC 541.13:541.64:547.898:546.3-128

#### Ion Transport Numbers in Crown-Containing Polymers

907M0158E Moscow IZVESTIYA AKADEMII NAUK  
SSSR: SERIYA KHIMICHESKAYA in Russian No 2,  
Feb 90 (manuscript received 12 Jan 89) pp 310-314

[Abstract of article by G. N. Altshuler and M. A. Khalyapina; Institute of Coal, Siberian Department, USSR Academy of Sciences, Kemerovo]

[Text] Transport numbers were measured for  $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Rb}^+$ ,  $\text{Cs}^+$ ,  $\text{Ti}^+$ , and  $\text{NO}_3^-$  ions migrating from electrolytes through homogeneous polymer-based membranes containing immobilized dibenzo-18-crown-6 and dibenzo-24-crown-8. Chlorides of the alkali metals and chemically pure  $\text{AgNO}_3$ , analytically pure  $\text{TiCl}_4$ , dibenzo-18-crown-6, and dibenzo-24-crown-8 were used. The polymer membranes were prepared by the polycondensation of dibenzo-18-crown-6 or dibenzo-24-crown-8 with formaldehyde and carefully rinsed with water and alcohol to remove the unlinked monomers. The working surface of the membranes was  $2.85 \text{ cm}^2$ . The Hittorf method was used to measure the transport numbers. A UIP-10 direct current supply was used. The density of the current passing through the membrane never exceeded  $0.40 \text{ mA/cm}^2$ . Current strength was measured by an R386 voltammeter from the drop in voltage on a standard calibrated resistor. Electrolyte concentration

on both sides of the membranes was determined: mercurimetrically for the  $\text{MCl}$ ; according to Folgard for the  $\text{AgNO}_3$ ; and for the  $\text{TiCl}_4$  by titration with a potassium bromate solution by using potentiometric control of the equivalence point. An EV-74 ion detector was used to control the pH level. It was shown that anions of the sorbed electrolytes evidently predominate in the transport of electrical charges in polymers. The role of the cations in the transport of electrical charges is diminished because of their coordination interaction with the crown-ethers in the polymer phase. Factors determining electrochemical mobility and ion transport numbers are the viscosity of the medium, the size of the migrating ions, and their interaction with the adsorption centers. Figures 2, tables 1; references 6: 4 Russian, 2 Western.

UDC 666.94:620.193.4

#### Hardening of Calcium, Strontium, and Barium Silicates and Their Solid Solutions in Corrosive Media

907M0204A Tashkent UZBEKSKIY KHIMICHESKIY  
ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript  
received 27 May 88) pp 3-5

[Article by R. A. Dzhakalayev, Z. P. Pulatov, and N. A. Sirazhiddinov; Institute of Chemistry, Uzbek SSR Academy of Sciences]

[Abstract] A method for synthesizing calcium, barium, and strontium silicates and their solid solutions was presented. Their hardening in magnesia-sulfate solutions was investigated, and the chemistry and mechanism of corrosive phenomena were studied. By analyzing the data obtained, the authors affirmed that the corrosion resistance of samples made of divalent metal silicates and their solid solutions depended primarily on the rate and amount of sulfate accumulation. References 5: 2 Russian, 3 Western.

UDC 666.016.1

#### Distrontium Titanodisilicate Formation Reaction

907M0204B Tashkent UZBEKSKIY KHIMICHESKIY  
ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript  
received 13 May 89) pp 5-7

[Article by B. Sh. Tuymetov and A. P. Irkakhodzhayeva; Institute of Chemistry, Uzbek SSR Academy of Sciences]

[Abstract] The process of distrontium titanodisilicate phase interrelation (by using chemical, x-ray diffraction, and thermographic analyses) and formation at  $900-1,300^\circ\text{C}$  was studied. It was established that the reaction was accompanied by the formation of interstitial strontium meta- and orthosilicate compounds. Distrontium titanodisilicate formation began at  $1,100^\circ\text{C}$  and was complete at  $1,300^\circ\text{C}$ . The glass began to crystallize at  $840^\circ\text{C}$ , and this was accompanied by the appearance of

interstitial compounds. Ditrontium titanodisilicate formed as a result. Figures 1; references 4: 3 Russian, 1 Western.

UDC 533.92

**Finely Dispersed Aluminum Oxide Powder  
Obtained at High Temperatures**

907M0227A Riga IZVESTIYA AKADEMII NAUK  
LATVIYSKOY SSR: SERIYA KHIMICHESKAYA  
in Russian No 2, Mar-Apr 90 (manuscript received  
13 Jun 89) pp 131-134

[Article by Ya. P. Grabis, I. P. Ubele, and A. B. Letlena;  
Institute of Inorganic Chemistry, Latvian SSR Academy  
of Sciences]

[Abstract] By means of vaporizing aluminum and its oxygen compounds in a high temperature flow, finely dispersed  $\delta$ - and  $\alpha$ - $\text{Al}_2\text{O}_3$  powders were obtained whose specific surface areas (20-70  $\text{m}^2/\text{g}$ ) were determined by the concentration of particles in the flow, the products' cooling conditions, and the composition of the starting powders. The temperature at which  $\alpha$ - $\text{Al}_2\text{O}_3$  began to form during finely dispersed aluminum oxide powder annealing decreased as the dispersion of the powder increased. Complete transition of the oxide to  $\alpha$ - $\text{Al}_2\text{O}_3$

occurred at 1350-1400°C and was preceded by an increase in grain size. Figures 4; references: 5 Russian.

UDC 666.112'92:543,544

**Structure of  $\text{K}_2\text{O-SiO}_2\text{-P}_2\text{O}_5$  Glass Systems**

907M0227B Riga IZVESTIYA AKADEMII NAUK  
LATVIYSKOY SSR: SERIYA KHIMICHESKAYA  
in Russian No 2, Mar-Apr 90 (manuscript received  
21 Jun 89) pp 161-164

[Article by S. Ye. Lagzdinya, I. A. Vitinya, U. Ya. Sedmalis, and D. K. Smildzinya; Riga Polytechnic Institute]

[Abstract] On the basis of IR spectroscopic and paper chromatographic investigations of  $\text{K}_2\text{O-SiO}_2\text{-P}_2\text{O}_5$  glass systems, it was established that the glass structure consists of linear and cyclic groups of  $\text{PO}_4$  tetrahedrons. Introducing  $\text{SiO}_2$  into this glass system in the ratio  $\text{K}_2\text{O:P}_2\text{O}_5 = 1:1$  led to depolymerization of the phosphate groups as well as in glasses with the ratio  $\text{K}_2\text{O:P}_2\text{O}_5 = 1:2$ . At a molar concentration of 5-10%,  $\text{SiO}_2$  acted as a binding element of linear phosphate groups and facilitated the formation of a more tightened glass structure. Figures 2; references 11: 9 Russian, 2 Western.

UDC 539.1.043:669.2925'295

**Structure and Phase Changes and Radiation Damage of Low-Activated Vanadium-Titanium Structural Alloys Subjected to Electron and Neutron Irradiation**

907M0282A Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 29 Jun 89) pp 5-11

[Article by L. I. Ivanov, V. M. Lazorenko, Yu. M. Platov, and V. I. Tovtin, Moscow]

[Abstract] There are prospects for the use of vanadium-titanium alloys in the walls of thermonuclear reactors. This is due to the rapid decrease of induced radioactivity in these materials, as well as their favorable construction and use properties, for example, high resistance to pore formation. The parameters of electron-positron annihilation (angular correlation), microhardness, and the structure of irradiated and nonirradiated vanadium and vanadium-titanium alloys were studied. It was found that microhardness varies monotonically with titanium content. The parameters of positron annihilation change sharply in the transition from pure vanadium to vanadium-titanium alloys. The parameters then remain virtually constant in the alloys. The angular correlation parameters show that alloying of vanadium with titanium increases the probability of positron annihilation on electrons, and this correlates with the change in electron density. In irradiated samples, the largest change in parameters of positron annihilation occurred in pure vanadium, which had a high concentration of radiation defects. The alloys also exhibited a change in the parameters of electron-positron annihilation. As the titanium content increases, the concentration of radiation defects decreases. Pore and void defects are also reduced in the alloys. Figures 5; table 1; references 12: 5 Russian, 7 Western.

UDC 621.039:620.17:669.292

**High-Temperature Mechanical Properties of Vanadium and Its Alloys Irradiated by Alpha Particles**

907M0282B Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 16 May 89) pp 12-16

[Article by Sh. B. Shiganakov, O. P. Maksimkin, L. I. Gomofov, S. N. Votinov, and B. G. Kurmanov, Moscow, Alma-Ata]

[Abstract] There are prospects for the use of vanadium as a construction material for thermonuclear reactors. However, the mechanical properties of vanadium irradiated by helium at high temperatures is virtually unstudied. The boundaries of grains and contaminants play a

large role in deformation and decomposition of semicrystalline material at high temperatures. Vanadium samples were manufactured and annealed in a vacuum. Some were then irradiated with 50-MeV alpha particles. Measures were taken to ensure uniform irradiation of the samples. The samples were then subjected to mechanical stress testing in a vacuum. The samples were held at the testing temperature (973, 1,073, 1,173 K) for 2 hours prior to testing. The deformations, surfaces, and microrelief of the samples were examined. Some phase separation was observed in all of the alloys. As temperature increased, the durability of the alloys, both irradiated and nonirradiated, decreased. The tensile strength and durability of the irradiated samples of all materials were somewhat lower than those of the nonirradiated samples. Irradiation effects were most clearly manifested in the plasticity of the samples tested at 1,173 K. The change in the high-temperature mechanical properties of vanadium alloys irradiated by alpha particles depends on the microstructure of the material, in particular, the state of the grain boundaries. The most durable and plastic alloys are those in which the grain boundaries are attached at some points to carbide particles. Figures 3; tables 2; references 8: 6 Russian 2 Western.

UDC 539.219.3

**Field-Ion Microscopic Studies of Mass Transport in Atomic Collision Cascades**

907M0282C Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 27 Jun 89) pp 17-19

[Article by V. T. Zabolotnyy, A. L. Suvorov, and V. P. Babayev, Moscow]

[Abstract] Atomic mixing is of interest as a means of creating pseudoalloys from components that have no noticeable mutual solubility in equilibrium conditions. This article proposes a method of using field-ion microscopy to measure the transport of mass by atomic collisions that mix and replace components. The parameters of damage produced by identical cascades of collisions in tungsten are used. The tungsten is irradiated at room temperature by 21-MeV electrons, 140-keV protons, 50-keV iron nuclei, and 50-keV tungsten ions. The principle of the method is to determine the difference in dispersion of the diffusant relative to some initial coordinate over the course of the experiment. At the center of the configuration is a region of increased void concentration. The average concentration of voids over the entire irradiated volume is significantly lower than the local concentration of voids in the depleted zone. It changes linearly over irradiation time. The experimental parameters of atomic mixing agree well with models proposed earlier. Figures 1; references 11.

UDC 669.2955.786

**Effect of Low-Temperature Nitrogen Implantation Into Substructure of Solid Alloys***907M0282D Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 9 Jan 89) pp 25-27*

[Article by Ye. I. Krichever, Kuybyshev]

[Abstract] It has already been shown that implantation may improve the durability and wear resistance of alloys. Nitrogen segregates to dislocations, blocking their movement and thus strengthening the alloy. This article examines the effect of implantation on the change in the substructure of the alloys Ti5Co10 and Ti15Co6. The size of the region of coherent scattering, the dislocation density, and microdeformations were studied. Their values were determined through approximation. Phase x-ray analysis was also used to determine the parameters of the crystal lattice of the alloys. The microhardness of the alloys was also studied. X-ray phase analysis showed that no new phases were formed over the entire range of doses studied. A nonlinear relation was found between the dislocation density, the relative increase in dislocation density, and the dose. The increase in microhardness relative to dose correlates well with the dose dependence of dislocation density. This is probably due to the instability of small angle boundaries for superequilibrium concentrations of point defects and radiation-stimulated diffusion. The depth of the hardened region reached 200-250  $\mu\text{m}$ . Microdistortions were completely absent over the entire range of doses. Figures 3; table 1; references 8: 6 Russian 2 Western.

UDC 669.295:621.785.54

**Explosion Hardening of Titanium Alloys***907M0282E Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 30 Mar 89) pp 43-48*

[Article by Ye. G. Popov, V. Z. Kutsova, N. V. Popova, A. G. Krimmel, and V. V. Sirostian, Dnepropetrovsk]

[Abstract] The explosion of compressed gas mixtures in a closed chamber can change the microstructure and phase content of metals to a depth of about 1 mm and thus harden the surface. Both single-phase heat-treated and two-phase untreated titanium alloys were tested in this way. The temperature of the detonation products reached 4,400 K, the pressure was about 100 MPa, and the reaction lasted about 0.1 seconds. The state of the samples after the explosion depended on the pressure and content of the mixture. Even in explosions at low pressures there was a noticeable increase in microhardness. At high pressures, inclusions began to appear. Surface etching revealed various characteristic zones of inclusions and varying microhardness levels. The surface layer of the titanium iodide sample exhibits zones of

lowered microhardness, and the inclusions that are clearly observed in the other titanium alloys are absent. Microroentgen analysis confirms the redistribution of titanium and alloy elements in the surface layer. The concentration inhomogeneities lead to the formation of several metastable phases. The products of the gas explosion have both a thermal and chemical effect on the samples due to alloying of the processed surface. Figures 5; tables 2; references 10.

UDC 539.4:533.915

**Effect of Low-Temperature Plasma on Surface of Tool Material***907M0282F Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 21 Feb 89) pp 49-52*

[Article by V. B. Trigub, I. V. Likholet, and M. A. Gaponov, Voronezh]

[Abstract] One promising method of hardening the surface of metals is plasma chemical deposition of coatings containing silicon. A hardness of 14,700-34,300 MPa may be obtained. The method is economical, quick, and easily regulated. It is more universal than vacuum coating is. The zone of the thermal effect is larger in the case of the plasma method than when the electron beam and laser methods are used in treating metal surfaces. This article studies the effect of various modes (times) of plasma chemical application of a hardening coating to the structurally stressed surface layer of a tool. The coating is applied in a low-temperature plasma flux at atmospheric pressure. Steel used for tools has high internal stress. Changes in this stress due to plasma chemical treatment can be examined in a diffractogram. Thus, the correct application time can be determined from the change in phase content and structural changes in the iron matrix. Application times must be chosen carefully for each type of steel, or weakening of the material can result. However, when the correct application time is chosen, the wear resistance of the material can be increased by a factor of 1.5-2.0. Figure 1; table 1; references 4.

UDC 620.197:621.357.8

**Formation Features and Several Properties of Coatings Obtained by Microarc Processing of Aluminum Alloys***907M0282G Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 9 Jan 89) pp 64-69*

[Article by V. S. Rudnev, P. S. Gordiyenko, A. G. Kurnosova, and M. V. Kovtun, Vladivostok]

[Abstract] Coatings obtained in microarc processing can protect metals from corrosion, mechanical wear, and the effects of heat. This article examines the effect of the

electrolyte, alloy, and processing time on the formation and electrophysical characteristics of coatings obtained in microarc processing. For identical conditions the electrolyte affects the change in voltage and the thickness of the coating. The thickness of the coating is dependent on the duration of the process, current density, and the composition of the electrolyte. In this experiment, the alloys have virtually no effect on the thickness of the coating. However, other authors have found some dependence on the alloy. Equations are developed for the thickness of the coating. Values are determined for the electric breakdown and resistance of the coating. Figures 4; tables 2; references 20: 13 Russian 7 Western.

UDC 539.23:621.793:535.211

**Laser Surface Alloying of Cold-Resistant Structural Steel To Increase Wear Resistance**

907M0282H Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 8 Jun 89) pp 70-75

[Article by M. L. Bernshteyn, A. M. Bernshteyn, and Ye. M. Brun, Moscow]

[Abstract] Cold-resistant two-phase steels are being successfully used instead of high-nickel steels in low-temperature conditions. It is desirable to treat the surface of these two-phase steels with a wear-resistant coating while maintaining the internal qualities of the material. A promising method of achieving this is laser surface alloying with armoring solid particles. Laser surface alloying is done by injecting vanadium carbide powder (plated with nickel) in a stream of inert gas into the surface layer of steel, which is melted by laser radiation. Laser surface alloying of steel (10Mn5Ni2MoVNb steel) with vanadium carbide produces a layer with a heterogeneous structure consisting of an austenite or austenite-martensite matrix with uniformly distributed armoring carbide particles of differing morphology and content. The hardest carbide-containing layers are obtained after tempering at 600-640°C with dispersion hardening and depletion of the austenite with carbon and alloy elements. The microhardness increases by 70-100% and is about 6,000 MPa. The surface layer obtained has a high relative wear resistance to dry friction. Figures 5; tables 2; references 4: 3 Russian 1 Western.

UDC 621.785.048:635.211

**Increasing Adhesion of Gas-Thermal Coatings Burnt Off by Laser Radiation**

907M0282I Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 7 Mar 89) pp 76-81

[Article by L. I. Grechikhin, N. V. Spiridonov, A. G. Vasilenko, A. G., M. A. Kardapolova, and O. G. Devoyno, Minsk]

[Abstract] A factor that limits the durability of the adhesion of gas-thermal coatings from self-fluxing alloys based on nickel and iron is the reduction of oxide films between the coating and substrate and the establishment of chemical bonds. A promising means of burning off gas-thermal coatings involves the addition of boron and silicon to reduce the oxide films. Lasers can be expediently used to localize treatment and to produce metastable structures. To increase adhesion one must completely reduce the oxide films. An optimum (minimal) processing time must be determined. Equations are developed to describe the kinetics of the reduction process. Processing time is dependent on the composition of the alloy and coating. Experiments to determine the durability of the adhesion of the coating to the substrate yielded results that correspond to the theoretical calculations. The durability of the bond was found to be close to that of a welded bond. Figures 4; table 1; references 6.

UDC 621.9.048.7

**Effect of Continuous Laser Radiation on Microhardness and Structure of Surface Layer of 20Cr2Ni4N Steel**

907M0282J Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 9 Mar 89) pp 82-84

[Article by V. P. Dmitriyev, Yu. N. Kalenikhin, and A. I. Sidorova, Gorkiy]

[Abstract] The effect of the radiation of a continuous-wave 1- to 1.5-kW CO<sub>2</sub> laser on 20Cr2Ni4N steel was studied. Variables in the experiment were the rate of movement of the sample relative to the laser beam and the amount of overlap of the laser tracks. The microstructure and the microhardness of the treated metal were tested, as well as the amount of radiation absorbed into the surface for a given treatment rate. The surface of the metal was treated with a protective but radiation-absorbing coating. Laser hardening can increase the microhardness of the metal by 25 percent in comparison to typical hardening methods. Increasing the speed of processing decreased the microhardness. It was determined that at least a 50% overlap of laser tracks results in a hardened zone without gaps. Figures 2; table 1; references 2.

UDC 669.15

**Study of Effect of Production Method on Optical Characteristics of Indium Films**

907M0282K Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 11 Apr 89) pp 89-91

[Article by S. N. Tarasenko, Novosibirsk]

[Abstract] Indium films may be used as ultraviolet filters for  $\lambda = 0.28 \mu\text{m}$ ; however, their optical properties vary depending on the production method. Laser and thermal methods of spray-coating are examined. Thermally produced films have a low transmission factor when compared with films produced by using laser methods. Inclusions in the thermally processed films increase absorption and scattering. The inhomogeneity of the film affects the amount of absorption and scattering. Inclusions and defects increase the coefficient of absorption. The films were tested for radiation resistance. Laser-produced films have a higher (by a factor of 20) threshold of radiation resistance. This is due to their lower coefficient of absorption. The transmission factor of laser-produced films is a factor of 20 greater than films that are thermally produced. Figures 2; references 3: 2 Russian 1 Western.

UDC 535.211:791.89

#### **Welding of Fittings for Pressurized Heavy Water Reactors. Laser as Alternative**

907M0282L Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 25 Jan 89) pp 106-112

[Article by G. L. Gosvami and Dilip Kumar, Bombay, India]

[Abstract] The fittings of fuel pipes in Indian pressurized heavy-water reactors are made of a zirconium alloy. The walls of the pipe are rather thin (0.4 mm). The weld must be durable, and the welding must not damage the pipe. Projection resistance welding is satisfactory, but the axial load on the joints is limited to 2,400 N. The load limit should be increased to 4,000 N. A laser welding method has been developed that may be successfully used for this purpose. A 300-W YAG laser was used. Welding was done in an argon-filled chamber with a

window for the passage of the beam. Argon was also passed through the interior of the pipe to prevent oxidation on the interior surface. New fittings were also developed that were better suited to the process of laser welding. Axial loads of 7,250 + 650 N could be supported by the laser welds. The entire periphery should be welded to obtain this level of load support. The welds and fittings were satisfactorily corrosion resistant. Figures 3; tables 4; references 2: 2 Western.

UDC 669.017

#### **Effect of Superplastic Deformation on Formation of Structure and Properties of Titanium Alloys**

907M0282M Moscow FIZIKA I KHIMIYA  
OBRABOTKI MATERIALOV in Russian No 3,  
May-Jun 90 (manuscript received 16 May 89)  
pp 120-123

[Article by Ye. B. Yegorov, V. N. Meshcheryakov, N. O. Yermakova, and S. P. Kulagin, Moscow]

[Abstract] Superplasticity can be effectively used as a component of thermomechanical processing of alloys, in particular, VT 6. Metal durability can be increased by increasing the density of dislocations and crystal structure defects. Accelerated cooling of an alloy after deformation makes this possible. Ordering of the dislocation structure increases its plasticity as well. The substructure of the metal is well developed. Grain and subgrain boundaries block the propagation of fissures. However, these fissures may branch. The propagation of microfissures in titanium alloys processed in a superplastic mode is similar to viscous decomposition. Thermomechanical processing of titanium alloys in superplastic modes is found to yield increased mechanical and use characteristics, thus increasing the reliability of the material in use and expanding its area of application. Figures 4; table 1; references 5.

UDC 542.91:543.422.25:547.1'118

**Interaction of Phosphorous Trichloride With Carboxylic Acids**

907M0158F Moscow IZVESTIYA AKADEMII NAUK  
SSSR: SERIYA KHIMICHESKAYA in Russian No 2,  
Feb 90 (manuscript received 7 Dec 88) pp 428-432

[Abstract of article by L. A. Valitova, Ye. V. Popova, Sh. N. Ibragimov, and B. Ye. Ivanov, Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan Affiliate, USSR Academy of Sciences]

[Text] The initial stages of  $\text{PCl}_3$  interaction with acetic, propionic, butyric, valeric, pelargonic, and lauric acids prior to oligomer formation were studied. The interaction of the phosphorous bottoms with propylene oxide and of phosphoric acid with propylene oxide was also studied. The following  $\text{PCl}_3$ -acetic acid interaction test served as an example of how the tests were conducted. A total of 41.25 g of  $\text{PCl}_3$  was added in portions during agitation to 54 g of  $\text{CH}_3\text{COOH}$ . The exothermic effect was 2-3°. Agitation for 2 hours at 45-50° yielded two layers, the bottom of which (28.1 g or 29.5%) was thrice rinsed with absolute petroleum ether and vacuum-evaporated. The upper layer was distilled off to yield 38.6 g (40.5%) acetyl chloride, b.p. 52-53°,  $n_D^{20}$  1.3870; 8.3 g (8.7%) acetic anhydride, b.p. 58-60° (50 mm),  $n_D^{20}$  1.3890; and 4.8 g (5%) acetic acid, b.p. 116-118°,  $n_D^{20}$  1.3710. NMR  $^{31}\text{P}$  spectra were taken on a KGU-4 with a working frequency of 10.2 Hz relative to 85%  $\text{H}_3\text{PO}_4$ . A Specord-75 IR spectrometer was used to record the IR spectra. The solvents used were dehydrated. The results showed that a mixture of P-H acids with a content of predominantly pyrophosphoric acid forms at 40-50°. The optimal reagent ratios were established from the acid chloride yields. Tables 2; references 15: 9 Russian, 6 Western.

UDC 542.97:541.63:547.26'118'161

**Catalytic Phosphorylation of Polyfluoroalkanols.  
14. Synthesis and Diastereoisomeric  
Transformations of Aryl( $\alpha$ -polyfluoroalkylbenzyl)  
chlorophosphates**

907M0158G Moscow IZVESTIYA AKADEMII NAUK  
SSSR: SERIYA KHIMICHESKAYA in Russian No 2,  
Feb 90 (manuscript received 20 Oct 88) pp 433-441

[Abstract of article by Ye. I. Goryunov, P. V. Petrovskiy, L. S. Zakharov, and M. I. Kabachnik; Institute of Heteroorganic Compounds imeni A. N. Nesmeyanov, USSR Academy of Sciences, Moscow]

[Text] Aryl( $\alpha$ -polyfluoroalkylbenzyl)chlorophosphates were synthesized by heating a mixture of 0.015 mol of  $\alpha$ -polyfluoroalkylbenzyl alcohol, 0.03-0.045 mol of aryldichlorophosphate, and 0.375 mmol of the appropriate catalyst for several hours at 140° until HCl liberation ceased; distilling off the surplus phosphorylation agent; and precipitating the final product in a vacuum from the distillation residue. The catalytic phosphorylation of p-methyl- $\alpha$ -trifluoromethylbenzyl alcohol (VI) with excess phenyldichlorophosphate (II) involved heating a mixture of 7.3 g (0.038 mol) of VI, 16 g (0.076 mol) of II, and 100 mg (0.9 mmol) of anhydrous  $\text{CaCl}_2$  at 130° for 5 hours until HCl liberation ceased. Individual diastereoisomers of the aryl- $\alpha$ -polyfluoroalkylbenzyl) chlorophosphates were then isolated. (Example: Two grams of phenyl( $\alpha$ -trifluoromethylbenzyl)chlorophosphate was twice recrystallized from hexane to obtain 0.7 g of diastereoisomer A [melting point, 83.5-85°].) PMR, NMR  $^{19}\text{F}$ , and  $^{31}\text{P}$  spectra were taken on a Bruker WP-200SY. Aryl( $\alpha$ -polyfluoroalkylbenzyl)chlorophosphates are formed during the catalytic phosphorylation of  $\alpha$ -polyfluoroalkylbenzyl alcohols. Control of the reaction stereochemistry is thermodynamic in nature, and the aryl( $\alpha$ -polyfluoroalkylbenzyl)chlorophosphates formed are equilibrium mixtures of diastereoisomers with a static 1:1 component ratio. The diastereoisomeric transformations proceeded in melt, in solution, and in the crystalline state, albeit in the opposite direction in the latter case. Figures 4, tables 4; references 5: all Russian.

UDC 665.61.033.55

**Characteristics of the Distribution of Vanadium, Nickel, and Their Porphyrin Complexes in Oil Components**

907M0237A Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 2, Mar-Apr 90 (manuscript received  
12 Jul 89) pp 170-174

[Article by R. A. Galimov, L. B. Krivonozhkina, V. V. Abushayeva, and G. V. Romanov; Institute of Organic and Physical Chemistry, Kazan]

[Abstract] The content of vanadium, nickel, and their porphyrin complexes in oil components of regular, sulfurous, and highly sulfurous oil and bitumen were investigated in order to establish general characteristics of these compounds' distribution. It was established that nickel porphyrins were concentrated in benzene tar, whereas vanadyl porphyrins were concentrated in phenol tar and asphaltenes. It is primarily nickel compounds that are concentrated in the benzene tar; they comprise 10-25% of the total tar content in the raw material. Between 30 and 50% of the nickel is in the form of porphyrin complexes, which account for 90% of all nickel porphyrins in each bituminoid. Vanadium accounts for between 20 and 35% of phenol tar, with 30-40% of this amount being in the form of porphyrin complexes and comprising 45-80% of the vanadyl porphyrin content in the oil. The nickel contribution was 10-20%. The asphaltenes were found to contain 50-80% nickel and vanadium, of which 7-10% is in the form of vanadyl porphyrins and accounts for 25-55% of the porphyrin complex content in the oil. References: 8 Russian.

UDC 665.61.033.55

**Microelement Composition of High-Molecular Weight Components in Azerbaijan Oil and Oil Residues**

907M0237B Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 2, Mar-Apr 90 (manuscript received 9 Oct 89)  
pp 175-183

[Article by G. N. Aleshin, F. I. Samedova, M. F. Mir-Babayev, and V. F. Kamyarov; Institute of Oil Chemistry, Tomsk; Tomsk Department of the Siberian Scientific Research Institute of Geology, Geophysics, and Mineral Raw Materials (SNIIGGiMS); Institute for Petrochemical Processes, Baku]

[Abstract] The concentrations of more than 20 metals (Fe, Ni, Co, V, Cr, Zn, Ba, Hg, Ag, La, etc.), bromine, and iodine were measured by the neutron-activation method in tars and asphaltenes taken from several typical Azerbaijani oils and from residues obtained from their distillation at >450°C. Iron series elements were found in the highest concentrations in the tar-asphaltene substances studied. The metal content decreased in the order Fe > Ni > Cr > V > Co. The predominant metal content in low-paraffin (naphthene) oil residues was concentrated primarily in the

asphaltenes, whereas in paraffin (methane, methanonaphthene) oil residues it was concentrated in the tar. In all cases, halogens were present primarily in tarry substances. During high-temperature vacuum distillation of the oil, the tar-asphaltene substances, as a rule, became enriched with Fe, Ni, Co, Cr, Zn, La, and Se atoms due to the accumulation of equipment corrosion products, and a significant portion of their original content of V, Ba, Hg, Ag, Br, and I atoms was lost. References 12: 11 Russian, 1 Western.

UDC 541.128

**Effect of Binary Solvent Composition on Rate of Nitrobenzene Hydration on Palladium Catalysts**

907M0237C Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 2, Mar-Apr 90 (manuscript received  
26 Dec 89) pp 195-197

[Article by A. A. Nasibulin, N. V. Sidorova, and M. V. Klyuyev; Ivanovo State University, Ivanovo]

[Abstract] The rate of nitrobenzene hydration on palladium catalysts in binary solutions of ethanol-DMFA, ethanol-hexane, and ethanol-pentanol generally decreased during the transition from ethanol to the mixtures. When using a swollen catalyst in the region of small additions of DMFA, hexane, or pentanol to ethanol, sharp changes were observed in the hydration rate due to changes in the amount of catalyst swelling. Figures 2; references: 2 Russian.

UDC 547.217.6:542.941.8:542.971.3

**Investigation of Magnesium-Vanadium Systems in the Process of Forming Olefins From *n*-Dodecane in Presence of Oxygen and Hydrochloric Acid**

907M0237D Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 2, Mar-Apr 90 (manuscript received  
4 Jan 90) pp 198-201

[Article by O. D. Sterligov, N. I. Rybakova, S. V. Adelson, and G. V. Isagulyants; Institute of Organic Chemistry imeni N. D. Zelinskiy, USSR Academy of Sciences, Moscow; Moscow Oil and Gas Institute imeni I. M. Gubkin]

[Abstract] The transformation of *n*-dodecane was studied at 500°C in the presence of oxygen, water vapor, and HCl on a magnesium-vanadium oxide catalyst and with magnesium and alkali sulfate additives. It was shown that the SO<sub>4</sub><sup>2-</sup> anion intensified the course of acid-base type reactions (cracking, skeletal isomerization). With the introduction of magnesium sulfate, *n*-dodecane conversion increased, the olefin yield increased due to the formation of C<sub>6-11</sub> olefins, and the overall olefin selectivity decreased almost by a factor of 2 (33%) due to intensified cracking reactions and skeletal isomerization. Adding alkali sulfates to the starting catalytic system somewhat increased the catalyst's activity, during which the C<sub>6-11</sub> and C<sub>12</sub> olefin yields were similar at an overall selectivity of 40-50%. Introducing hydrochloric acid to the reaction medium



facilitated increased activity in the dehydrogenation reaction (by a factor of 1.5-2) due to the formation of a significant amount of *n*-dodecenes. On the samples with lithium or sodium sulfate additives, the yield of C<sub>6-12</sub> olefins reached 15% with a selectivity of 89%. It was proposed that dehydrogenation of the starting hydrocarbon into *n*-dodecenes occurred without the participation of oxygen and, particularly in the experiments with HCl, according to the oxidative dehydrogenation reaction. References 12: 10 Russian, 2 Western.

UDC 547.491.6.057:542.91:665.7.038

**Producing Dialkyl Cyanamides—Depressor Additives for Diesel Fuels**

907M0237E Moscow NEFTEKHIMIYA in Russian  
Vol 30 No 2, Mar-Apr 90 (manuscript received  
27 Jul 89) pp 257-264

[Article by Yu. N. Polivin, V. V. Yurechko, T. P. Vishnyakova, and Ye. A. Ageyev; Moscow Oil and Gas Institute imeni I. M. Gubkin]

[Abstract] With the aim of determining the optimum conditions for synthesizing dialkyl cyanamides with long aliphatic radicals, i.e., depressor additives for diesel fuels, the reaction of potassium cyanamide with the halogenated alkyls *n*-C<sub>4</sub>H<sub>9</sub>Cl, *n*-C<sub>4</sub>H<sub>9</sub>Br, *n*-C<sub>4</sub>H<sub>9</sub>I, *sec*-C<sub>4</sub>H<sub>9</sub>Br, *tert*-C<sub>4</sub>H<sub>9</sub>Br, *n*-C<sub>12</sub>H<sub>25</sub>Br, *n*-C<sub>8</sub>H<sub>17</sub>Br, *n*-C<sub>15</sub>H<sub>31</sub>Br, and *n*-C<sub>16</sub>H<sub>33</sub>Br in isopropanol, methanol, and dimethyl formamide was studied. Dialkyl cyanamides could be synthesized in the highest yields in the dipolar aprotic solvent, i.e., dimethylformamide (more than 90%). In this solvent, as opposed to isopropanol and methanol, alcoholysis of the starting salt, which reduces the dialkyl cyanamide yield, did not occur. References 20: 13 Russian, 7 Western.

UDC 547.73:541.11.547.898.057

**First Example of Synthesis of Stable Potassium Chlorodiperoxychromate in Complex Form With Crown Ethers and Study of Thermal Stability***907M0128B Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 35 No 1, Jan 90 (manuscript received 3 Apr 89) pp 92-96*

[Article by G. V. Fedorova, N. V. Novozhilova, L. S. Fedorova, G. L. Tudorovskaya, S. A. Kotlyar, and N. G. Lukyanenko, Monomer SRI (A-U), Tula; Physical Chemistry Institute imeni A. V. Bogatskiy, Odessa]

[Abstract] Hexavalent chromates and bichromates of sodium or potassium have recently found applications as mild oxidizing agents in organic syntheses taking place in nonpolar solvents or quaternary ammonium salts. Recently, a complex of potassium bichromate and a crown ether has been prepared, but there are still no references in the literature on complexes of hexavalent chromium peroxy compounds with crown ethers or their properties. In the present work stable complexes of the title compound with 15-crown-5, 18-crown-6, and dicyclohexane-18-crown-6 were prepared. Composition and structures were confirmed by various means such as element and emission analysis and IR and UV spectroscopy. Thermal decomposition and intermediate and end products were studied by x-ray and differential thermal analysis. Figures 2; references 15: 5 Russian, 10 Western.

UDC 541.64:539.2

**Structure of Polyethylene Gel-Fiber***907M0129A Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 13 Jun 88) pp 8-13*

[Article by Ye. P. Krasnov, L. D. Rudneva, Yu. I. Mitchenko, A. N. Dyachkov, V. I. Kuzub, and A. S. Chegolya, Synthetic Fibers SRI (A-U), Kalinin

[Abstract] The development of super-strong and elastic fibers from polyethylene is one of the major recent achievements of polymer science. In an essentially new method for shaping fibers, a hot solution of super-high molecular weight PE (over  $10^6$ ) is shaped directly in water. The latter, being immiscible with the solvent, acts as an effective coolant. Supercooling the solution causes phase separation and formation of a gel still filled with the original solvent in which the polymer is no longer solvent at room temperature. After drying, the resulting gel has a many-fold stretch factor with high strength and elasticity. Formation of a gel from super-high molecular weight PE is a specific feature of this process and there is reason to assume that this stage offers potential possibilities for super-stretching and marked increases in strength and other properties of the fiber. In the present work mercury porosimetry was used to determine the total

pore size of super-high molecular weight PE gel-fiber and its structure. Contractual phenomena, occurring during vaporization of included liquids, have a marked effect on the structural characteristics of the gel-fiber. The gels are uniform in volume and have a cell-honeycomb configuration. Even slight deformation stress in the gel system can cause straightening of the molecular chain. Figures 2; references 10: 7 Russian, 3 Western.

UDC 541(64+127):542.952

**Effect of Forming Mechanism of Polymer-Monomer Particles on Kinetics of Emulsion Polymerization of Acrylic Monomers***907M0129B Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 16 Jun 88) pp 14-19*

[Article by I. A. Gritskova, Ye. B. Malyukova, G. A. Simakova, and V. P. Zubov, Fine Chemical Technology Institute imeni M. V. Lomonosov, Moscow]

[Abstract] Acrylic acid latexes are versatile products finding applications in the medical, textile, paper, and other industries as adhesives and impregnating agents. However, carrying out emulsion polymerization and copolymerization of acrylic monomers presents problems due to low emulsion stability and formation of coagulums in the end product. Although this can be offset to some extent by procedures such as component fractionation, this tends to complicate the technology of the process. To simplify preparation of latexes with single batch loading of components to the reaction mix while maintaining high emulsion stability, the formation mechanism of the polymer-monomer particles must be known. In the present work, a study was made of colloidal chemical properties of methyl, ethyl, and butyl acrylate monomer emulsions as prepared with both ionic and non-ionic emulsifiers. These emulsions have highly dispersed monomer macro-droplets and also contain micro-emulsion drops. Emulsion polymerization of the monomers in the presence of various surfactants demonstrated that monomer micro-droplets have a significant effect on the formation of polymer-monomer particles. Figures 6; references 9: 7 Russian, 2 Western.

UDC 541.64:539.2

**Polyester Urethanes from 1,5-Naphthalene Diisocyanate. Structure and Thermal Behavior***907M0129C Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 4 Jul 88) pp 25-30*

[Article by B. Ya. Teytelbaum, A. T. Gubaydullin, T. A. Yagfarova, N. P. Apukhtina, and L. A. Cherkasova (deceased), Organic and Physical Chemistry Institute imeni A. Ye. Arbuzov, Kazan]

[Abstract] 1,5-Naphthalene diisocyanate is frequently associated with the synthesis of polyester urethanes, the presence of condensed aromatic rings being a favorable factor in the formation of rigid segments in the polymer chains of thermoplastics. The structure of these materials is assumed to have rigid domes interconnecting flexible polyester chains into a single "physical" network. Polyurethanes synthesized from 1,5-naphthalene diisocyanate have not yet been studied from this standpoint. In the present work polyester urethane thermoplastics were synthesized from oligobutylene adipate having a molecular weight of about 2000, 1,5-naphthalene diisocyanate, and 1,4-butanediol with various ratios of functional groups. Wide and small angle X-ray diffraction confirmed the preservation of crystallinity of the polyurethane segment during disintegration of the domain structure at temperatures exceeding 200°. The data further demonstrated that polymer flow is not a result of dome melting or mutual solubility of phases, but rather the outcome of thermal dissociation of interdomain urethane bonds. The crystal field stabilizes both the urethane groups and the hydrogen bonds within the domes. An excess of NCO groups during synthesis enhances the thermal stability of the polymer. Figures 5; references 17: 14 Russian, 2 Western.

UDC 541.64:539.199:547.458.82

#### Hydrodynamic Properties and Equilibrium Rigidity of Oxypropyl Cellulose Molecules

907M0129D Moscow VYSOKOMOLEKULYARNYYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90  
(manuscript received 21 Jul 88) pp 43-48

[Article by Ye. V. Korneyeva, I. N. Shtennikov, V. P. Shibayev, S. I. Klenin, G. F. Kolbina, I. V. Yekayeva, and S. A. Didenko, High Molecular Compounds Institute, Leningrad]

[Abstract] Interphase formation is currently widely studied in oxypropyl cellulose, an ester capable of displaying mesomorphic properties in the molten state and in various solvents. A thermotropic cholesteric mesophase has been observed in oxypropyl cellulose melts at 160-205°. Analysis of these phenomena requires information on the molecular parameters of oxypropyl cellulose. In the present work a study was made of the intrinsic viscosity, translational diffusion, and sedimentation of samples and fractions of oxypropyl cellulose in various solvents. These properties were then correlated with the degree of polymerization, and the equilibrium rigidity of the macromolecule was determined from translational diffusion and viscosimetric data. The possibility of mesophase formation in melts and solutions is apparently due to the high rigidity of the macromolecule. Figures 6; references 19: 9 Russian, 8 Western.

UDC 541(183.12+64):546.212

#### Interactions of Carboxylic Hetero-Net Polyelectrolytes with Water

907M0129E Moscow VYSOKOMOLEKULYARNYYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90  
(manuscript received 22 Jul 88) pp 61-65

[Article by V. S. Yurchenko, K. P. Papukova, and G. V. Samsonov, High Molecular Compounds Institute, Leningrad]

[Abstract] Copolymerization of methacrylic acid with a long chain grafting agent such as N,N'-ethylenedimethacrylamide (EDMA) in acetic acid solutions results in the formation of density heterogeneous polymers at a certain EDMA content. Macronet polymers formed with low EDMA content have a low number of physical nodes, but high swelling and polymer permeability due to the large celled structure of the network. At high EDMA content density heterogeneous copolymers are formed which are porous only in the swollen or dry states. The thickening-thinning dimensions of the polymer net in N,N'-ethylenedimethacrylamide copolymer are only ten or more nanometers, and an increase in the ratio of thinned matrix components as a result of increased grafting agent content leads to an extreme relationship between water sorption and grafting agent content for heterogeneous polymers. In the present work it was demonstrated that the integral free energy of water vapor sorption on the above copolymer system in H-form increases proportionately with EDMA content to an absolute value. Figures 4; references 10: 7 Russian, 3 Western.

UDC 532.77:541.64:539.2

#### Structural-Rheological Processes During Fiber Preparation from High Molecular Polyethylene Solutions

907M0129F Moscow VYSOKOMOLEKULYARNYYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90  
(manuscript received 25 Jul 88) pp 77-82

[Article by V. G. Kulichikhin, M. Kh. Mirdzhanov, Ye. M. Antipov, Yu. I. Mitchenko, Ye. V. Popova, V. I. Kuzub, and S. A. Kuptsov, Petrochemical Synthesis Institute imeni A. V. Topchiev, Moscow]

[Abstract] The preparation of high strength fibers from semi-dilute solutions of flexible-chained polyethylene (molecular weight exceeding 10<sup>6</sup>) has attracted much attention. Compared to using melts, these systems make it possible to lower network density by solution, fix the structure by cooling, purge the solvent from the crystallized system, and stretch the resulting xerogel to the extent required for high molecular orientation and physical-mechanical specifications of the finished fiber. In the present work it was observed that semidiluted super-high molecular polyethylene solutions have a high degree

of structurization resulting in viscous-plastic behavior, and the level of longitudinal stress during xerogel stretching is a function of the shear stress under which the extrudate was formed. Stress-deformation curves, derived during stretching, may be divided into orthorhombic and pseudo-hexagonal phases. Figures 6; references 15: 6 Russian, 9 Western.

UDC 541.64:543.422.4

### Long Wave IR-Spectra and -Relaxation in Glassy Polymers

907M0129G Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 25 Jul 88) pp 90-95

[Article by V. A. Ryzhov, V. A. Bershteyn, and A. B. Sinani, Physical Technical Institute imeni A. F. Ioffe, Leningrad]

[Abstract] Relaxation phenomena are frequently observed in flexible chained glassy polymers at 80-170 K, which is intermediate in respect to  $\beta$ -transition and low temperature  $\delta$ -transition. In the present work long wave IR-spectra were used to study  $\gamma$ -relaxation in several oligomers, linear and moderately grafted polymers. Absorption data obtained at the 120-170  $\text{cm}^{-1}$  range may be used to help clarify the molecular mechanism of  $\gamma$ -relaxation. Except for some special cases, this transition in flexible glassy polymers appears to be the result of limited torsional vibrations in segments of the backbone significantly shorter than the statistical segments. Figures 4; references 28: 12 Russian, 16 Western.

UDC 541.64:533.15

### Selectivity and Permeability in Amorphous Polymer Membranes

907M0129H Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 1 Aug 88) pp 127-131

[Article by A. V. Matveyev and M. N. Tulsikiy]

[Abstract] The well known advantages of membrane separation of gases is offset by high costs as compared to traditional methods of gas separation. Membrane technology may be made more competitive through development of highly productive and selective polymers. Experimental data indicate that the selectivity of a membrane in any single class, is inversely proportional to its productivity. In the present work theoretical formulas derived from a lattice model of a polymer were used to analyze the selectivity and permeability of gas separating amorphous polymer membranes. It was observed that the sub-molecular structure and solubility parameters of the polymer affect the gas separating characteristics of the membrane. Figures 3; references 15: 10 Russian, 5 Western.

UDC 541.64:539.3

### Molecular Nature of Reversible and Irreversible Deformation in Flexible Chain Polymers

907M0129I Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 1 Aug 88) pp 132-135

[Article by P. M. Pakhomov, V. P. Nanasnikov, M. V. Shablygin, and A. S. Chegolya, Synthetic Fibers SRI (A-U), Kalinin]

[Abstract] Both reversible (elastic) and irreversible (plastic) deformations occur simultaneously in a polymer during stretching. Plastic deformation may result from mutual sliding of molecular chains (physical flow) or molecular ruptures (chemical flow). The relationship between the plastic and elastic components of deformation at a given temperature depends on the load method and duration of mechanical action. Total deformation may be separated into its components by relaxation (settling) of stretched polymer samples after relieving the load. In the present work polarized IR-spectroscopy was used to correlate the accumulated fraction of elastic deformation with the conformational composition and degree of orientation of molecular chains, and the accumulated fraction of plastic deformation with the concentration of molecular ruptures in samples of polyethylene film. Figures 4; references 11 (Russian).

UDC 541.64:535.5:539.199

### Application of Sinusoidal Pulses in Kerr Effect to Study Dynamics of Polymer Molecules in Conducting Solutions

907M0129J Moscow VYSOKOMOLEKULARNYE SOYEDINENIYA in Russian Vol 32 No 1, Jan 90 (manuscript received 10 Mar 89) pp 162-165

[Article by A. V. Lyezov and N. V. Tsvetkov, Physics Institute at Leningrad State University]

[Abstract] Electrical double refraction, or the so-called Kerr effect, has been shown to be an effective means for studying conformational, dynamic and electro-optical properties of rigid polymer molecules in solution. However, rigid polymers, as a rule, are only soluble in strong polar solvents which have both high conductivity and Kerr effect. Therefore, Kerr effect studies under these conditions is possible only by using a pulsed technique. By using square wave pulses with direct current voltage and elliptic polarization modulation of the light bundle, it is possible to measure the Kerr effect in polymer solutions using solvents such as acetone, cyclohexane, or dichloroacetic acid. Although these measurements make it possible to determine equilibrium Kerr effect values, the basic question of the nature of the Kerr effect, i.e. is it caused by rigid or induced molecular dipoles, can only be resolved by using an alternating pulsed field. However, such measurements have not yet been made in

highly conducting solutions. In the present work a technique was developed for measuring Kerr effect in conductive solutions using pulsed sinusoidal fields at various frequencies. As an example, results are presented on solutions of aromatic polyamidobenzimidazole. The data contain information on both dynamic and equilibrium electro-optical characteristics of this polymer. Figures 3; references 6 (Russian).

UDC 66.095.268+66.018.83+543.87

### **Irradiation Hardening of Oligoestermaleate-Styrene Compositions**

907M0149D Kiev UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 56 No 1, Jan 90 pp 88-92

[Abstract of article by S. A. Bratslavskaya, N. G. Videnina, S. I. Omelchenko, V. A. Machtin, A. V. Sokolov, and Ye. M. Pliss]

[Abstract] Oxidation processes occurring during thermochemical and irradiation-chemical hardening of film-forming oligomer-monomer compositions were studied to determine how the inhibiting effect of atmospheric oxygen is affected by certain additives. The compositions consisted of oligoethylenepropylenemaleatephthalate, styrene, and a number of additives: acrylic acid, cellulose acetobutyrate, and 2,4,6-tris(dimethylaminomethyl)-phenol or its methylated analogue 2,4,6-tris(dimethylaminomethyl)-methoxybenzene, all of which help to reduce the inhibiting effect of oxygen during polymerization. All of the compounds were purified by multiple low-pressure reprecipitation or distillation. Initiated oxidation was studied volumetrically from the oxygen absorption rate. Radicals were generated thermochemically (azodi-isobutyronitrile initiator at 343 K). The solid and viscous substances were oxidized in chlorobenzene purified by a previously described method. The hydroperoxide concentration was determined iodometrically. The inhibiting effect of oxygen on the polymerization of these compositions was noticeably reduced. The explanation for this is that during hardening intense hydroperoxide formation occurs in the surface layer as the cellulose acetobutyrate oxidizes. The aminophenol and acrylic acid cause these hydroperoxides to quickly decompose, and this process in turn leads to a higher rate of radical formation in the surface layer. These mobile, highly active radicals interact intensively with the unreacted  $\pi$ -bonds of the molecules and with the alkyl radicals, which, being highly concentrated in the surface layer, basically enter into a chain-termination reaction that helps to form a cross-linked polymer. Figures 2, tables 2; references 18: 16.Russian, 2 Western.

UDC 678.065.002.2:001.892

### **Main Directions in Research and Development To Improve Tire Production Technology**

907M0162A Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 2-7

[Abstract of article by Yu. P. Bass]

[Abstract] A major goal of the tire industry is to improve tire quality and durability. All aspects of tire production need to be mechanized and automated. Computer models of tire production should be developed and applied. Quality control should focus on the quality of individual tire components and production steps as well as on the final product. Design should drive tire production technology. New tire designs and tire production technologies must be adopted. Other related objectives are to increase labor productivity 2- to 3-fold, reduce energy consumption 1.5- to 2-fold, improve working conditions, and pollute less. As part of the effort to improve the rubber stock, automated continuous mixing units with capacities of 630 and 370 liters and circulatory heat-exchange systems will come on line within the next few years. Researchers are studying the following: the relationship between mixing equipment design and the kinetics of the formation of the structure and properties of rubber stocks, energy requirements in light of hydrodynamics and heat exchange, adhesive-frictional and other properties, hard-to-process rubber stocks, and the use of powder rubbers. Cord fabric can be improved by increasing rubberization uniformity and by heating the cord with an air and combusted gas products mixture. The delivery of instruments for determining the surface density of rubberized cord is scheduled to begin this year. A nonconventional rubberization method, impregnation after thermal stretching, is under development. Work is also being done to improve steel cord rubberization uniformity and the strength of the bond between the metal and the rubber stock. Ways to improve component preparation and assembly operations include breaking them down into individual automated jobs done at specialized work stations, protecting tire components by doing assembly work in climate-controlled facilities, using artificial light, etc., and determining the type and amount of equipment needed to convey specific components. Vulcanization also needs to be improved through automation, the use of more sophisticated equipment, and tighter process controls. References 28: 24 Russian, 4 Western.

UDC 678.065.054

### **Main Directions and Outlook for Production of Tire-Making Equipment From 1995 to 2000**

907M0162B Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 7-11

[Abstract of article by B. M. Petrov]

[Abstract] The Soviet tire industry is working to automate all aspects of the conventional tire-building process, integrate the various assembly steps into single production lines, make greater use of flexible manufacturing systems and automated conveyance systems, and incorporate new and better processes, materials, and equipment. The DRA 2—0—45° bias cutting machine is under development. It has two material feed stations and is slated for production in 1990. The DRA 3—0—45°

has three feed stations and can be programmed to cut three types of rubber cord sequentially into 4,500-mm strips. It is slated for production in 1991. Microprocessor control and +1-mm cutting accuracy are 1995 goals for both machines. The AKD 80/1300 ring-forming machine, which has been in production since 1987, outperforms the AKD 70/1300 by 20-40%. The AKD 2 320/620 has a 1.7-fold greater productivity. The AK 650-1500 is used for making bead rings from steel ribbon for 39- to 45-inch tires, and the SKR 1000-1500 is used for making beads from these rings. The Scientific Research Institute of Tire Making Equipment [NIIshtinmash] is refining the capability to cold-mold radial car tire beads with a 70-mm filler cord height in order to double and greatly improve quality. The AKS 400-600, which is microprocessor controlled, is programmed to make bead rings with complex cross-sections from a single wire. The AKR 500-600 (currently under development) is intended for making beads with high filler cord for 20- and 22.5-inch steel-construction truck and auto tires. The Yaroslavl Tire Plant is learning to use equipment that can produce eight beads for 20-inch truck tires with filler cord up to 20 mm high at the same time; plans now exist to start up three-station units for making car tire beads with high filler cord. Also under development is an automated machinery system that is intended for use in automating all aspects of body construction. It is being used at the Bobruyskshin Production Association and is available for delivery to other firms without such integrated systems. The SPP 3-460-800, which has PS 460-800 conveyers for assembling 15- to 16-inch bias tires is microprocessor controlled and comes equipped with a high-speed drum collapser, tread rubber joint molder, "gapless" precision drums, and tread rubber feeders. Plans have been formulated to draft the design documents for building 13- to 14-inch and radial tires on these machines, which will be able to feed two fabric plies from either reels or boggles. The ASPR 360-600 (13-14 inch) and ASPR 440-660 (15-16 inch) (i.e., the first Soviet-built four-position tire assembly unit), are intended for use in building steel- and composite-belted radial car tires with single- and double-ply bodies, open or closed beads. The NIIshtinmash and NIIShP plan to increase their productivity to 32.1 units/h and to equip them with belt-strip feeders and improved feed systems. New equipment for assembling 205R14 and 135R12 tires is being made. ASPRs will increase productivity 1.7- to 1.8-fold by requiring only one operator. The Bobruyskshina Production Association is learning to run an industrial prototype automated machinery system with 9 industrial robots and 32 autoloading, which can increase productivity 2.6-fold and replace up to 100 workers at an output of 900,000 tires/yr (20 sec/tire). The NIIshtinmash forecasts a demand of 5 to 10 such systems in the 13th Five-Year-Plan. The SPD-2 and SPD-3 are microprocessor-controlled standardized machine tools with high-speed drum collapsers that have been modernized for radial tire assembly; about 40 SPD 2-660-900 MIs with 15-20% higher productivity will be produced in 1990. They will increase labor productivity by a factor of 1.5 to

1.7. The LSPR 710-1150, LSPR 2-710-1100, and LSPR 660-950 lines (for building the bodies of a wide assortment of 200- 508R to 320-508R-size radial truck tires) have been automated to increase labor productivity, reliability, and durability. Between 1996 and 2000, they will be replaced by new-generation lines such as the LSPR 2-510-485, which will be used in conjunction with computer-controlled modular lines with automated conveyance systems to increase labor productivity 1.8- to 2.0-fold in comparison with individual IDO- 59s and SPRI-2Ms. By the year 2000, 40 FMS will be in use in the tire industry and will increase labor productivity 3- to 3.5- fold and replace up to 9,000 workers. The LSPRM 550-1000, which has four assembly stations, is intended for use in building 20- to 22.5-inch steel-cord tires, a critical problem for the tire industry. It has a projected productivity of 18.5 or 24 tires/h, depending on belt construction, and it is being used at the Yaroslavl Tire Plant along with the ASPRM 550-1000 for building 20- to 22.5-inch steel-cord truck tires. Included among the improved oversized and agricultural tire-building equipment are the SPPR 1300 (to replace the SPPR 1100-1400), the SPD-4, and the ASPR system. Advances made by foreign tire companies, particularly Firestone, Goodyear, and Dunlop, and their importance to the Soviet tire industry are also discussed.

UDC 678.05"71":678.065

#### State of Tire-Making Equipment and Prospects for Development

907M0162C Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 12-14

[Abstract of article by A. G. Posternak]

[Abstract] The author contends that the tire industry could improve its performance greatly by taking advantage of equipment and processes currently available or under development. In the area of rubber compounding and extrusion equipment the All-Union Scientific Research Institute for Rubber-Processing Equipment [VNIIRTmash] and the Bolshevik Scientific Production Association [NPO] have built the ATK-1 automated production line (capacity, 16 t/h) and the ATK-3 (capacity, 8 t/h) for making master and finished rubber stock. The ATK-3 is better than foreign-made equipment at processing stiff stock. An ATK consisting of a 270/15...60 mixer, an AChVL-600 extruder, and a festoon machine with an AF-6N-600 adjustable drive is being constructed. The VNIIRTmash has made the first prototypes of machines for making rubber stock from powdered or crumbled rubbers. In the area of tire stock-making equipment, the VNIIRTmash has developed the LPL- 800 and LBL-800 tread and sidewall lines equipped with cold-feed extruders and modern local mechanization and automation devices to replace IRU-16, 592-16, and Krupp tread lines; a line for making truck tire ply liners from two types of rubber; and lines for making tubes from general-purpose and butyl-rubber

compounds. It is building a line with two extruders sharing a common head and automated process control for producing precision tread and sidewall stock. The LOK-80-1800, APK-80-1800, and ATK-80-1800 are recommended for rubberizing cord. The LOK-80-1800 has a new four-roll calender with individual roll drives, devices for widening the cord edges, and an automatic device for parting the rolls as the seam passes through. The LOK-87-1800, with an MChKh cold-feed extruder for feeding the calender instead of rollers, is being developed for the Moscow tire plant and will automate and mechanize most steps in the rubberizing process. The LPTK-87-1800 mechanized line with automated process control for rubberizing and heat-treating cord is being developed for the Volga tire plant; delivery is scheduled for 1992. It will use about 1.5 times less energy and metal. This type of equipment is slated for delivery to tire plants during the 13th and 14th Five-Year-Plans. As far as curing equipment is concerned, existing vulcanization and curing equipment is outmoded and must be replaced. Soviet-made equipment is being modeled after foreign-made equipment, which is fully mechanized and automated, is microprocessor-controlled, has mandrel-equipped curing molds and zonal heating, and is capable of exerting 2.8 MPa of pressure in the diaphragm. Experimental models include the FV 2-120, 2-160, 2-300, and 1-150 and the VPMS 2-120, 2-160, and 2-200, all of which have electromechanical drives except for the FV 2-300, which has a hydraulic drive supplied from the shop main. Production of an experimental FVSVCh 1-360 for making KI-80 fabric-based casings is planned for 1990. It should increase productivity 1.5- to 2-fold by using very high frequency current to heat the green tire from the diaphragm side and electric heaters to heat it from the mold side. The Scientific Research Institute for Tire Production [NIIShP] is working to develop high-priority liquid-molding (oligomer compositions) technology. Experimental models of the LRL-125/63 and ARL-16/630-4 rotary-type molding equipment for making tire valves and solid tires, respectively, are planned for 1991. Despite these advances, the industry is having difficulties making mandrel-equipped molds and microprocessor-based equipment. In the area of tire-retreading equipment, the production of modernized I-90GMU and I-170GMU vulcanizers was established in 1988 at the Dnepropolimer mash Plant. This was done to replace outmoded GM-type, Marangoni, and Scoda equipment. The I-230GMU was tested in 1988 and has been recommended for series production. The VNIIRT-mash and the Bolshevik NPO are working together to design more reliable and better-performing 2-90GMR, I-170GMR, and I-230GMR vulcanizers with rod-and-crank drives for the upper platform. The new vulcanizers will come with steam-, water-, or air-driven power plants, depending on the needs of the customer. Work is also being done to develop and improve equipment for recycling used tires and waste from tire production, such as the MIP-1 and LRK-320.

UDC 678.065

### Improvements in Technology and Equipment for Manufacturing Tire Components

907M0162D Moscow KAUCHUK I REZINA in Russian  
No 1, Jan 90 pp 16-18

[Abstract of article by A. I. Terekhov, I. M. Muslayev, I. A. Spivak, and Ye. B. Kipnis]

[Abstract] Improvements in tire production equipment and technology during the 11th and 12th Five-Year-Plans were surveyed. Improvements in the DRA 0-45° bias-cutting machine were described and include a cutting accuracy of +1.5-2 mm for the strip width and +0.5° for the angle, a rotary blade driven by a cutting bogie drive, a feed reel that can accommodate wider rolls of fabric, a safe and easy-to-use device for splicing the fabric, and an improved control system with data-processing and light and sound signaling capabilities. Work is being done to make the linear asynchronous motor drive of the cutting bogie more reliable, and experimental models have been introduced. Existing equipment has been modernized by changing the way it is arranged. For example, the 921-01 lay-up machine for two-sided lay-up of the rubber ply liners was rearranged so that the 3-330-500 calender faces the main lay-up calender. Before, the body plies had to pass through the machine twice in order to apply the liners to both sides of the body, and this led to stretching and buckling of the fabric strips. Now the liners can be applied in one pass, thereby improving body ply quality and labor productivity. Incorporation of automated process control and the use of specialized as opposed to universal equipment has also increased productivity and conserved energy and materials. Optimal arrangement of lines for manufacturing the body ply stock for car and truck tires was described and illustrated. The quality of the bead rings has been improved by using AKDs incorporating MChKh-63 and MChKh-90 cold-feed extruders. A prototype of the AKS 400-600 has been built and is slated for series production. It is designed to make complex-shaped bead rings two at a time for 20- and 22.5-inch tires. Pirelli bead-construction machines will remain in use until the year 2000. Figures 3, references 32: Russian.

UDC 678.065.001.2-52

### Automating Tire Research and Design: Current State, Problems, and Outlook

907M0162E Moscow KAUCHUK I REZINA in Russian  
No 1, Jan 90 pp 26-27

[Abstract of article by V. N. Moskalenko]

[Abstract] Existing systems for tire research and development are the first-generation Tire Design CAD System [SAPR-Sh] and Automated System for Tire Research

[ASNI-Sh]. The SAPR-Sh consists of a PDP 11/34 computer with terminals and graphics devices or a YeS-1033 with terminals and printers. It is used to design radial tires and has five subsystems to calculate design parameters, carry out tire stress and strain experiments, perform data processing, and execute graphics. The ASNI-Sh is set up on a computer network of SM-1420 computers with remote terminals and three SM-1300-based control computer systems, which are connected to the test benches. It is designed to support the manufacturing and testing aspects of tire design and has two subsystems for automating experiments and providing data in support of ongoing research. Organizational, methodological, and technical bugs still need to be worked out, however. The main organizational problem is that these systems are unable to handle the flood of orders for automatic tire design, which necessitates greater testing and data processing capacities. There are a host of methodological problems ranging from tire design theory to the application of mathematical models to increase standardization and professionalism in research methods to the establishment of data banks. The primary technical problems are a lack of the necessary computer equipment and networks and the inability to tie into city-, nation-, and worldwide data banks. A comprehensive theory of automated tire design encompassing all stages of a tire's life cycle is being worked out. Efforts continue to upgrade computer systems at research organizations and throughout the industry. An industry-wide computer network and data base should be established as soon as practicable.

UDC 678.065.001.2

#### Basic Design Considerations for Low-Profile Truck and Bus Tires

907M0162F Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 27-28

[Abstract of article by V. P. Timofeyev, M. I. Rekitar, and V. N. Prashchikin]

[Abstract] Research was done to determine the optimal section dimensions for series 70 and 80 low-profile truck and bus tires. For series 70 tires,  $H/B = 0.70-0.72$ , relative tread width was increased to 0.90, and relative section width was increased to 110% of ordinary-profile tire section width. For series 80 tires, these dimensions are 0.82-0.83, 0.80-0.85, and 105%, respectively. For both tire series, relative rim width and tread curvature radius are 0.70-0.75 and 2.6-3.0, respectively. The formulas used to calculate these dimensions were provided. These data were used to design the 310/80R508 and 370/80R508 HP-54/56 tires. The 310/80R508 is replacing the double 260R508 ordinary-profile tires on KamAZ truck trailers and semi-trailers, and the 370/80R508 is to be used on KAZ agricultural tractor-trailers. Figures 2, table 1; references 5: Russian.

UDC 678.4.065:629.11.012

#### Current State of, Problems in, and Outlook for Development of Large and Superlarge Tires

907M0162G Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 29-31

[Abstract of article by A. G. Nechiporenko, A. G. Smirnov, and A. A. Lapkina]

[Text] In the area of large and superlarge tires, the Soviet tire industry needs to develop heavy-duty steel-body and steel-belted radial tires (18.00R25, 21.00R33, 21.00R35, 24.00R35, 33.00R51, and 40.00R57 for quarry vehicles; 11.00R20, 14.00R20, 17.5R29, 18.00R25, and 21.00R33 for mining equipment; and 14.00R20 for road-building equipment); improve the design of special-purpose bias-ply tires; develop a basic inventory of advanced-design tubeless tires; use new and better reinforcing materials to reduce materials consumption and improve performance characteristics; improve durability by using better, thermally stable tread rubbers applied by winding; increase the inventory of specialized solid high-elasticity tires; and bring tire performance and quality up to world-class levels. In comparative tests with conventional bias-ply tires, 18.00R25 and 21.00R33 tires were shown to have superior durability, load-carrying capacity, tractive and speed characteristics, and fuel economy. Materials that need to be developed include steel cord with a breaking strength up to 20 kN, high-modulus synthetic fibers and high-quality natural rubbers, carbon black, and chemical additives. When used in place of 23KNTS cord, 30A cord, production of which is limited, increases wear resistance 8-10% and average tire life 5-8% while reducing mass 8-10%. The industry also needs to develop equipment for rubberizing and cutting steel cord, tire-building machines and vulcanizers, and accessory equipment for producing these types of tires. Greater use of flexible production systems is needed to accommodate the ever-growing variety of specialized tires. Cordless solid highly elastic 4.00—8, 18X7—8, 650—10, 7.00—12, and 355/65—15 "superelastic" tires for ordinary and heavy-duty transport vehicles are under development. The industry also needs to establish greater research, experimental production, and testing capabilities. References 14: 12 Russian, 2 western.

UDC 678.01:629.11.012.533

#### Developing Tread Rubbers With Enhanced Performance Properties for Future Tire Designs

907M0162H Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 32-37

[Abstract of article by N. L. Sakhnovskiy, L. I. Stepanova, E. A. Anfimova, and T. A. Koroleva]

[Abstract] The results of a number of studies on the relationship between the performance properties of tread



rubbers and their composition, strength, and elastic hysteresis properties and on the interrelation between the latter and structure are summarized. Research has shown that elastic hysteresis properties determine basic performance characteristics. Optimal elasticity, as measured by stress at 300% elongation ( $f_{300}$ ), depends on tire construction, driving conditions, and the type of polymer used in the rubber. In general, increasing elasticity results in greater resistance to wear and decreased rolling losses, but it can also cause a higher incidence of tread failures. It is recommended that  $f_{300}$  be increased as much as practicable without inducing cracking and crumbling in the tread rubber. Concerning the relationship between structure and properties, it has been determined that elastic strength characteristics are determined by the structure of the vulcanization network (cross bonds, polymer chain modification, etc.). Recommendations for the following are presented: optimize vulcanization and phase interaction by using fewer plasticizers and dispersing the filler as completely as possible; tailor tread rubber specifications to tire purpose and road conditions; do more research on the relationship between structure and properties and look for additional ways to improve the vulcanization network; develop new tread-rubber compounding materials; and develop a variety of truck, bus, and car tread rubbers maximizing wear resistance, traction on wet and icy surfaces, and fuel economy while minimizing hysteresis and rolling losses. By the year 2000, wear resistance is expected to increase 30%, losses are expected to drop 10%, and traction on wet and icy surfaces is to be substantially improved. Figures 6, tables 7; references 7: 6 Russian, 1 western.

UDC 678.01:678.046.2:677.53

**Influence of Type of Carbon Black Used on Properties of Coating Rubbers for Brass-Plated Steel Cord**

907M0162I Moscow KAUCHUK I REZINA in Russian No 1, Jan 90 pp 37-38

[Abstract of article by L. T. Goncharova, G. D. Minayeva, V. L. Maloyenko, and Ye. A. Okhotnikova]

[Abstract] Different types of carbon black (N234, N220, N375, N347, N330, S315, N326) were added to rubber compounds based on 100% SKI-3. The different types of carbon varied according to particle size, degree of structure, surface state of oxidation, and ability to adsorb steam. The mixtures also contained two modifying systems (parts by weight): I, 2 parts RU + 5 parts BS-120; II, 1.5 parts RU + 5 parts BS-120 + 1 part cobalt naphthenate (10% Co content). The viscosity and adhesive properties of the compounds, the elasticity and strength of the rubbers made from them, and the bond strength between the steel cord and the coating rubber were then measured and compared. It was shown that the strength of the bond between the steel cord and the coating rubber

is greatly affected by the choice of carbon black and modifying system used in a particular compound. Tables 3; references 2: Russian.

UDC 541(49+64):547

**Grafted Cellulose Acetate-Polymethacrylic Acid Copolymer Interpolymer Complex With Polyvinylpyridine Oxide**

907M0204G Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 9 Dec 88) pp 36-38

[Article by F. F. Nurgaliyeva, N. I. Kurbanova, and R. S. Tillayev; Tashkent Order of the Red Banner of Labor State University imeni V. I. Lenin]

[Abstract] The properties of aqueous solutions of grafted cellulose acetate (water-soluble)-polymethacrylic acid copolymers of varying composition and their polycomplexes with polyvinylpyridine oxide were studied by potentiometric titration. It was shown that the grafted copolymers underwent conformational changes during the ionization process, whereas conformational conversions were not detected for their polycomplexes. Figures 3; references 6: 4 Russian, 2 Western.

UDC 541.64+677.494.745:532

**Molecular Weight Distribution of Acrylonitrile Copolymers With Vinyl Monomers as Function of Process Temperature**

907M0204H Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 26 Jan 89) pp 39-40

[Article by A. A. Mamazhanov, A. S. Boymirzayev, and Sh. A. Fuzailov; work completed under the direction of Corresponding Member of the Uzbek SSR Academy of Sciences M. A. Askarov; Institute of the Chemistry and Physics of Polymers, Uzbek SSR Academy of Sciences]

[Abstract] The viscosity, molecular weight, and molecular weight distribution of the ternary acrylonitrile copolymer with methylacrylate and acrylic acid in dimethylformamide were studied. It was established that the temperature factor had an inversely proportional effect on the molecular weight of the forming acrylonitrile copolymer. This was explained by the fact that higher temperatures increased the rate of all elementary stages of copolymerization. Increasing the concentration of radicals and the rate of radical polymerization led to the formation of smaller macromolecules, i.e., a polymer with a lower molecular weight. Figures 1; references: 3 Russian.

UDC 541.64:547.39

**Photopolymerization of Butylmethacrylate With Acrylonitrile in Presence of Quinoline-Bromide Complex***907M0204I Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 2 Dec 88) pp 40-42*

[Article by Z. S. Sabirov, N. I. Iskhakov, and Kh. R. Akhmedova; work completed under the direction of Corresponding Member of the Uzbek SSR Academy of Sciences M. A. Askarov; Tashkent Order of the Peoples' Friendship Institute of Textile and Light Industry imeni Yu. Akhunbabayev]

[Abstract] The photopolymerization of butylmethacrylate (BMA) with acrylonitrile in the presence of a quinoline-bromide complex was investigated, and the properties of the resultant pre-copolymers were studied. It was shown that in the presence of a quinoline-bromide complex, one could obtain butylmethacrylate pre-copolymers with acrylonitrile that exhibit an adequate molecular weight (ranging from  $4 \cdot 10^5$  to  $10^6$ ). Figures 1; references: 3 Russian.

UDC 541.64:678(-13+743)

**Dielectric Relaxation in Vinyl Fluoride Copolymers With Fluoroethylenes***907M0204J Tashkent UZBEKSKIY KHIMICHESKIY ZHURNAL in Russian No 1, Jan-Feb 90 (manuscript received 9 Dec 88) pp 42-45*

[Article by N. I. Yakubov, A. Kh. Gafurov, M. U. Akhrayeva, and M. Sh. Salikhova; Tashkent Order of the Red Banner of Labor State University imeni V. I. Lenin]

[Abstract] Molecular mobility in vinyl fluoride copolymers with vinylidene fluoride, trifluoro-, chlorotrifluoro-, and tetrafluoroethylene was studied in the range 175-450 K. It was shown that the copolymers' dielectric relaxation depended on the structure of the monomeric units and on their intramolecular distribution in the chain. Increasing the microblock character of the macromolecules as well as changing the intra- and intermolecular interaction apparently led to the onset of kinetic mobility of segments located in the high elasticity region with a simultaneous reduction in dielectric losses. Figures 2; references 5: 4 Russian, 1 Western.